BOILER HOUSE MERCURY INVESTIGATION REPORT
AK STEEL
KANSAS CITY, MISSOURI

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NOV 19 2012
AWMD/WRAP-KNRP

USEPA ID NO.: MOD007118029

NOVEMBER 2012

Prepared for

AK Steel

Prepared By

Burns & McDonnell Project No. 69635
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Engineers-Architects-Consultants
Kansas City, Missouri

RCRA
521439

AK Steel Corporation
Environmental Affairs
9227 Centre Pointe Drive
West Chester, Ohio 45069

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NOV 19 2012
AWMD/WRAP-KNRP

November 15, 2012

Mr. Bruce Morrison, Project Manager
Waste Remediation and Permitting Branch
Air and Waste Management Division
United States Environmental Protection Agency - Region VII
11201 Renner Boulevard
Lenexa KS 66219

Re: HSWA Corrective Action Permit Number MOD 007 118 029
Draft Boiler House Mercury Investigation Report
AK Steel, Kansas City, Missouri

Dear Mr. Johnson:

AK Steel is submitting to the Environmental Protection Agency (EPA) and the Missouri Department of Natural Resources (MDNR) the *Draft Boiler House Mercury Investigation Report,* which was prepared by Burns & McDonnell Engineering Company, Inc. (Burns & McDonnell) at our direction. This report was developed to present the results of investigation activities conducted in accordance with the *Quality Assurance Sampling and Analysis Plan for the Boiler House Mercury Investigation* (BMcD, 2012).

CERTIFICATION:

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision according to a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

This Report and Certification are submitted on behalf of AK Steel Corporation.

Very truly yours,

James C. Levengood
Corporate Manager of Environmental Affairs

cc: C. Kump-Mitchell – MDNR (1 Copy)
B. Stuart – MDNR (2 Copies)
C. Batliner – AK Steel
S. L. Shelton – Burns & McDonnell

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LIST OF ACRONYMS AND ABBREVIATIONS

Burns & McDonnell	Burns & McDonnell Engineering Company, Inc.
DQI	data quality indicator
ft bgs	feet below ground surface
Facility	AK Steel, 7000 Winner Road, Kansas City, Missouri
GST	GST Technologies Operating Co., Inc.
I-435	Interstate 435
mg/kg	milligrams per kilogram
MS	matrix spike
MSD	matrix spike duplicate
NELAP	National Environmental Laboratory Accreditation Program
NFGI	*Contract Laboratory Program National Function Guidelines for Inorganic Superfund Data Review*
ng/m^3	nonograms per cubic meter
NRC	USEPA National Response Center
Pace	Pace Analytical Services, Inc.
QA	quality assurance
QA SAP	*Quality Assurance Sampling and Analysis Plan for the Boiler House Mercury Investigation*
QC	quality control
RSL	Regional Screening Levels
SEP	Sequential Extraction Procedure
SOP	Standard Operating Procedure
USEPA	United States Environmental Protection Agency

DOCUMENT DISTRIBUTION

USEPA Region 7, Bruce Morrison, Project Manager – 2 copies

Missouri Department of Natural Resources, Christine Kump-Mitchell, Project Manager – 1 copy

Missouri Department of Natural Resources, Bruce Stuart, Sr. Technical Advisor – 2 copies

AK Steel, Cory Levengood – 1 copy

AK Steel, Carl Batliner – 1 copy

Burns & McDonnell Engineering Company, Inc., Sharon Shelton – 2 copies

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1.0 INTRODUCTION

On February 2, 2012, the United States Environmental Protection Agency (USEPA) National Response Center (NRC) received an anonymous report of alleged historical mercury dumping associated with GS Technologies Operating Company, Inc. (GST) operations in the 1990s[1]. According to the NRC report, a total of 125 to 300 pounds of surplus mercury was allegedly buried under the floor of a boiler room. A copy of the NRC report is provided in Appendix A. The NRC report resulted in representatives of USEPA visiting the AK Steel Kansas City Works (Facility) on February 3, February 17, and June 8, 2012 and being provided access to a former Boiler House and the former Ball Department Boiler Room (see Appendix B). Based on the NRC Report and mercury vapor screening performed in the two areas, USEPA requested that AK Steel submit a work plan for additional sampling of the former Boiler House to verify the results of the mercury vapor screening. In August 2012 the *Final Quality Assurance Sampling and Analysis Plan for the Boiler House Mercury Investigation* (QA SAP) was submitted on behalf of AK Steel by Burns & McDonnell Engineering Company, Inc. (Burns & McDonnell) in response to USEPA's request.

1.1 PURPOSE AND SCOPE

This *Boiler House Mercury Investigation Report* presents the results of the surface material sampling within the former Boiler House at the AK Steel Facility located in Kansas City, Missouri. Figure 1-1 provides a Facility Location Map, and Figure 1-2 depicts the Facility layout.

These activities were performed to collect the data requested by the USEPA based on the June 8, 2012 mercury vapor screening. Data collection activities included mercury vapor screening and characterization of total mercury in surface materials within the former Boiler House. Sample collection methodologies for surface materials, including sampling requirements for quality assurance (QA)/quality control (QC) programs were summarized in the QA SAP (Burns & McDonnell, 2012).

[1] Historically, the plant operations and property owned by Armco (currently AK Steel) totaled approximately 860 acres. GST purchased approximately 300 acres of property in 1993 and also leased approximately 100 acres. GST operated on this property until they filed for bankruptcy in April 2001. The anonymous reporter alleged that the mercury dumping occurred on property under the control of GST.

1.2 BACKGROUND

1.2.1 Facility Location

Figure 1-1 presents a Facility Location Map. The Facility is located in northeast Kansas City, Missouri within the Blue River and Missouri River floodplains. Portions of the Facility are located both east and west of Interstate Highway 435 (I-435). Industrial activities were performed exclusively in the area west of I-435, north of 12th Street, and east of Ewing Avenue. Figure 1-2 depicts the Facility and presents ownership and operational changes that have occurred since issuance of the Permit, and Figure 1-3 depicts the former Boiler House. The current address for the AK Steel Kansas City Facility is:

AK Steel
7000 Winner Road
Kansas City, Missouri 64125

1.2.2 Facility History

The Facility history was previously described in Section 2.2.2 of the QA SAP (Burns & McDonnell, 2012).

1.2.3 Permit History

The permit history for the Facility was previously described in Section 2.2.3 of the QA SAP (Burns & McDonnell, 2012).

1.2.4 Environmental Setting

The environmental setting for the Facility was previously described in Section 2 of the RFI Report (BMWCI, 1999).

1.3 REPORT ORGANIZATION

This *Boiler House Mercury Investigation Report* has been prepared by Burns & McDonnell and consists of one volume. This document is organized as follows:

- Section 1.0 – Introduction
- Section 2.0 – Introduction to the Data Presentation
- Section 3.0 – Surface Material Sampling at the Former Boiler House
- Section 4.0 – Summary and Conclusions
- Section 5.0 – References

* * * * *

2.0 INTRODUCTION TO THE DATA PRESENTATION

During the surface material investigation, samples were collected for chemical analyses based upon verification field screening in accordance with the QA SAP. Section 3.0 presents the findings for the field screening and sampling of surface materials in the former Boiler House. Fourteen surface material samples (including one field duplicate) were collected and submitted for laboratory analysis of total mercury.

General supporting information for the data and text provided in this *Boiler House Mercury Investigation Report* is provided in the following Appendices:

- Appendix C – QA/QC Review of Analytical Data
- Appendix D – Field Logbook
- Appendix E – Analytical Laboratory Reports

2.1 DATA ANALYSIS

2.1.1 Quality Control Evaluation

Pace Analytical Services, Inc. of Lenexa, Kansas (Pace) provided laboratory services for the surface material sampling within the former Boiler House. Pace is certified as part of the National Environmental Laboratory Accreditation Program (NELAP).

The laboratory data were reviewed for achievement of QA/QC criteria. Field QC samples included a field duplicate, matrix spike/matrix spike duplicate (MS/MSDs), and a temperature blank. Data quality indicators (DQIs) that were evaluated include: precision, accuracy, representativeness, comparability, and completeness. Data verification and validation were performed following procedures outlined in the QA SAP. Data qualifiers, when appropriate, were added to the data in accordance with *USEPA's National Functional Guidelines for Inorganic Superfund Data Review* (NFGI) (USEPA, 2010). A description of data qualifiers assigned by the analytical laboratories and during data verification and validation are provided in Appendix C.

Based upon the results of the data verification and validation, the data were considered valid to use in reporting the results of the surface material sampling at the former Boiler House. As indicated in the QA/QC Review of Analytical Data (Appendix C), the objectives for precision, accuracy, representativeness, completeness, and comparability were met.

2.1.2 Screening of Data

Screening levels are used to determine the nature and extent of contamination and may subsequently serve as action levels for various activities. Surface material samples were analyzed for total mercury using USEPA method SW-846 7471 (USEPA, 2008). Following analysis, surface material total mercury results were screened using the USEPA Regional Screening Level Table (RSL) for industrial soil (USEPA, 2012) for elemental mercury of 43 milligrams per kilogram (mg/kg). Since there is the potential of mercury gauges previously present within the Boiler House to have been vandalized or damaged, the USEPA RSL for elemental mercury was selected rather than the RSL for mercuric chloride (and other mercury salts). The discussion in Section 3.0 includes a comparison of sample results to the screening level. Constituents that exceed screening levels are highlighted in the data summary table.

2.2 DATA PRESENTATION

Analytical data are presented in analytical results data tables and in text discussions. Figures are provided to indicate mercury vapor screening results and surface material sampling locations within the former Boiler House.

* * * * *

3.0 SURFACE MATERIAL SAMPLING AT THE FORMER BOILER HOUSE

3.1 BACKGROUND AND SUMMARY OF PREVIOUS ACTIVITIES

On February 2, 2012, the USEPA NRC received an anonymous report of alleged historical mercury dumping associated with GST operations in the 1990s. According to the NRC report, a total of 125 to 300 pounds of surplus mercury was allegedly buried under the floor of a boiler room (see Appendix A). The NRC report resulted in representatives of USEPA visiting the Facility on February 3 and February 17, 2012 to inspect former boiler areas, including the former Boiler House and Ball Department Boiler Room (See Appendix B).

On June 8, 2012, USEPA returned to the Facility to conduct mercury vapor screening of the former Boiler House and Ball Department Boiler Room using an Ohio Lumex Mercury Analyzer. Results for the two areas were as follows:

- Boiler House – Background reading for mercury vapor collected from outside of the building ranged from 30 to 38 nanograms per cubic meter (ng/m^3). Mercury vapor readings were similar to background in the area of disturbed soil/rubble inside the northwest part the building. Mercury vapor readings greater than 2,000 ng/m^3 were noted between several concrete footings assumed to be former equipment mounts along the south wall of the building. In this same area, mercury vapor readings greater than 2,000 ng/m^3 were also noted adjacent to the intact boiler where gauges appear to have been mounted. A shallow layer of soil, broken up building foundation materials, and other debris were noted in this area.

- Ball Department Boiler Room - Background readings of mercury vapor in this area averaged 56 ng/m^3. None of the readings inside the Ball Department Boiler Room exceeded this value.

3.2 SCOPE OF ACTIVITIES COMPLETED

Field activities were conducted on September 26, 2012. Field measurements of mercury vapor in ambient air were recorded using the Ohio Lumex RA-915 monitor prior to sample collection. An initial set of background reading measurements were collected from the parking area on the west side of the former Boiler House (Figure 1-3) and used for comparison to samples collected within the Boiler House. Mercury vapor readings were recorded at various locations within the former Boiler House to identify surface material sample locations (Figure 3-1). According to the QA SAP, ten sample locations were planned based upon mercury vapor reading results. However, the USEPA Project Manager was on-site during field activities and requested additional sampling locations based upon the field screening results.

Therefore, 13 surface material sample locations (Figure 3-2) were collected from within the Boiler House at locations confirmed with the USEPA Project Manager. The sample collection summary is outlined on Table 3-1. Surface material samples were collected from 0.0 to 0.5 feet below ground surface (ft bgs), and consist of primarily erosional deposits and deteriorated anthropogenic surface materials. Mercury vapor readings were also recorded at each sample location during sample collection (Table 3-2). Surface material samples were collected and submitted to Pace for analysis of total mercury using USEPA Method SW-846 7471. Field QC samples included one field duplicate and one MS/MSD pair.

3.3 INVESTIGATION RESULTS

The initial mercury vapor readings that were recorded prior to determining surface material sample locations are presented on Figure 3-1. The mercury vapor readings collected from the surface material in the sample containers and analytical results for the surface material samples from within the former Boiler House are presented on Table 3-2. Figure 3-3 presents the surface material sample locations and associated off-site analytical results for total mercury. Mercury detections were screened against the USEPA RSL for Industrial Soil for elemental mercury (43 mg/kg).

Total mercury was detected in all surface material samples ranging from 1.9 mg/kg to 275 mg/kg. Samples BHSM09/SS 0-0.5 (275 mg/kg) and BHSM12/SS 0-0.5 (83.6 mg/kg) exceeded the 43 mg/kg industrial soil screening level for elemental mercury. Pace diluted these samples by factors of 500 and 250, respectively, which could introduce uncertainty into the total mercury quantitation. Sample BHSM09/SS 0-0.5 was located on the south side of the existing boiler, and Sample BHSM12/SS 0-0.5 was located near the bay opening along the south wall of the building. All other surface material sample detections were below the industrial soil screening level for elemental mercury. When comparing mercury vapor readings recorded at the sample location and from the sample jars to analytical results for total mercury, no correlation between the values can be drawn.

* * * * *

4.0 SUMMARY AND CONCLUSIONS

On February 2, 2012, the USEPA NRC received an anonymous report of alleged historical mercury dumping associated with GST operations in the 1990s[2]. According to the NRC report, a total of 125 to 300 pounds of surplus mercury was allegedly buried under the floor of a boiler room. The NRC report resulted in representatives of USEPA visiting the Facility on February 3, February 17, and June 8, 2012 and being provided access to a former Boiler House and the former Ball Department Boiler Room. Based on the NRC Report and mercury vapor screening performed in the two areas, USEPA requested that AK Steel submit a work plan for additional sampling of the former Boiler House to verify the results of the mercury vapor screening. In August 2012 the QA SAP was submitted that outlined investigation activities, and a one-day sampling event was conducted on September 26, 2012. The purpose of this *Boiler House Mercury Investigation Report* is to present the mercury sampling results within the former Boiler House at the AK Steel Facility located in Kansas City, Missouri.

Field measurements of mercury vapor in ambient air were recorded using the Ohio Lumex RA-915 monitor prior to surface material sample collection. Background readings were recorded outside the Boiler House, and mercury vapor screening was performed throughout the building to identify potential sampling locations (Figure 3-1). Based upon the field screening results and consultation with the USEPA Project Manager who was on-site during sampling activities, surface material samples were collected from 13 locations (Figure 3-2) using procedures outlined in the QA SAP.

Mercury vapor readings were recorded from each sample jar during sample collection (Table 3-2). Surface material samples were submitted to Pace for analysis of total mercury using USEPA Method SW-846 7471. The mercury vapor readings at surface material sample locations and the analytical results for surface material sample results collected from within the former Boiler House are presented on Table 3-2. Figure 3-3 presents the surface material sample locations and associated off-site analytical results for mercury. Sample results were screened against the USEPA RSL for Industrial Soil for elemental mercury (43 mg/kg).

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[2] Historically, the plant operations and property owned by Armco (currently AK Steel) totaled approximately 860 acres. GST purchased approximately 300 acres of property in 1993 and also leased approximately 100 acres. GST operated on this property until they filed for bankruptcy in April 2001. The anonymous reporter alleged that the mercury dumping occurred on property under the control of GST.

Total mercury was detected in all surface samples at concentrations ranging from 1.9 mg/kg to 275 mg/kg (Table 3-2). Samples from 11 of the 13 locations exhibited concentrations below the USEPA RSL. Samples BHSM09/SS 0-0.5 (275 mg/kg) and BHSM12/SS 0-0.5 (83.6 mg/kg) exceeded the 43 mg/kg USEPA RSL for Industrial Soil for elemental mercury (43 mg/kg). Sample BHSM09/SS 0-0.5 was located on the south side of the existing boiler, and Sample BHSM12/SS 0-0.5 was located near the bay opening along the south wall of the building (Figure 3-3). There was no apparent correlation between the mercury vapor screening results and the associated total mercury analysis for the samples. That is, mercury vapor readings taken from sample jars that were orders of magnitude above background did not necessarily correlate to elevated total mercury results in the off-site analysis of the surface material.

Due to the limited number of exceedances of the elemental mercury RSL and a lack of correlation between the Ohio Lumex Meter mercury vapor field screening results and the total mercury results, confirmation sampling is proposed for locations BHSM09 and BHSM12. Procedures described in the QA SAP (BMcD, 2012) will be used for sample collection and associated field activities. Samples will be submitted to Brooks Rand Labs of Seattle, Washington for analysis of the following constituents:

- Total mercury – USEPA SW-846 7471. This analysis will be performed to provide an indication of the total amount of mercury present in the sample, regardless of species (i.e., The results include mercury salts, organic mercury, and elemental mercury).

- Mercury Speciation – Brooks Rand Labs Standard Operating Procedure (SOP) BR-0013, Five-Step Selective Sequential Extraction Procedure (SEP) to Quantify Mercury Factions in Sediments, Soils, and Mine Tailings. This procedure is described in Appendix F. This analysis will be performed to provide the types of mercury species present in the sample, as follows:

Fraction	Description	Extractant	Typical Compounds
F1	Water Soluble	Distilled Water	$HgCl_2$ $HgSO_4$
F2	Weak Acid Soluble/"Stomach Acid"	pH 2 Hydrochloric Acid/Acetic Acid	HgO
F3	Organo Complexed	1M Potassium Hydroxide	Hg-humics Hg_2Cl_2 CH_3Hg
F4	Strong Complexed	12M Nitric Acid	Mineral lattice Hg_2Cl_2 Hg^0

Fraction	Description	Extractant	Typical Compounds
F5	Mineral Bound / Cinnabar	Aqua Regia	HgS m-HgS HgSe HgAu
In addition, Brooks Rand Labs will perform a pre-extraction step, referred to as F0. This is a qualitative presence/absence analysis where the sample is purged to obtain volatile mercury. Volatile mercury includes unbound elemental mercury and dimethyl mercury. Dimethyl mercury is typically only detected in environmental samples in certain landfill gases. Since the F4 fraction includes both elemental mercury and other species, the presence of mercury in the F0 analysis is an indication that mercury detected in the F4 fraction is likely elemental.			

A sample collection summary is provided on Table 4-1, and a summary of analytical methods, containers, preservatives is provided on Table 4-2. The point of contact for Brooks Rand Labs is as follows:

Name	Primary Point of Contact
Brooks Rand Labs 3958 6th Avenue NW Seattle, WA 98107	Elizabeth Madonick phone: (206) 632-6206, ext. 141 email: elizabeth@brooksrand.com

Based on the mercury species identified during sampling, the data will be compared to the appropriate USEPA RSL for industrial soil (USEPA, 2012), as follows:

- Mercuric chloride (and other mercury salts): 310 mg/kg
- Mercury (elemental): 43 mg/kg
- Methyl mercury: 100 mg/kg

Sample collection activities are planned to be completed approximately 30 days following approval of the proposed additional sampling by USEPA and MDNR. Submittal of letter report summarizing supplemental investigation results is planned approximately 60 days following completion of sample analysis.

* * * * *

5.0 REFERENCES

Burns & McDonnell, 2012, *Final Quality Assurance Sampling and Analysis Plan for the Boiler House Mercury Investigation for AK Steel, Kansas City, Missouri.* August.

Burns & McDonnell Waste Consultants, Inc., 1999. *RCRA Facility Investigation Report, Armco Kansas City Facility.* September.

USEPA, 2008. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. Final Update IV.* January.

USEPA, 2010. *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review.* January.

USEPA, 2012. *Regional Screening Levels (RSLs) Summary Table.* May. Available from: http://www.epa.gov/region9/superfund/prg.

* * * * *

TABLES

Table 3-1
Surface Material Sample Summary
Boiler House Mercury Investigation Report
AK Steel Facility - Kansas City, Missouri

Sample Point	Sample Designator & Point	Sample Type	Estimated Depth of Sample (ft bgs)	Mercury (SW846 7471)
BHSM01	SS0-0.5		0.0 - 0.5	X
BHSM02	SS0-0.5		0.0 - 0.5	X
BHSM03	SS0-0.5		0.0 - 0.5	X
BHSM04	SS0-0.5		0.0 - 0.5	X
BHSM05	SS0-0.5		0.0 - 0.5	X
BHSM06	SS0-0.5		0.0 - 0.5	X
BHSM07	SS0-0.5		0.0 - 0.5	X
BHSM08	SS0-0.5		0.0 - 0.5	X
BHSM08A	SS0-0.5	Duplicate	0.0 - 0.5	X
BHSM09	SS0-0.5		0.0 - 0.5	X
BHSM10	SS0-0.5		0.0 - 0.5	X
BHSM10MS/MSD	SS0-0.5	MS/MSD	0.0 - 0.5	X
BHSM11	SS0-0.5		0.0 - 0.5	X
BHSM12	SS0-0.5		0.0 - 0.5	X
BHSM13	SS0-0.5		0.0 - 0.5	X

Notes:
ft bgs - feet below ground surface
MS - matrix spike
MSD - matrix spike duplicate

Table 3-2
Surface Material Sample Results
Boiler House Mercury Investigation Report
AK Steel Facility - Kansas City, Missouri

Sample ID	Laboratory ID	Collection Date	Total Mercury SW-846 7471 (mg/kg)	Mecury Vapor Field Screening[1] (ng/m^3)
BHSM01/SS 0-0.5	60129815001	9/26/2012	**8.7**	1198 - 1224
BHSM02/SS 0-0.5	60129815002	9/26/2012	**13.1**	1992 - 2467
BHSM03/SS 0-0.5	60129815003	9/26/2012	**2.8**	1249 - 2390
BHSM04/SS 0-0.5	60129815004	9/26/2012	**1.9**	955 - 1562
BHSM05/SS 0-0.5	60129815005	9/26/2012	**2.1**	641 - 2020
BHSM06/SS 0-0.5	60129815006	9/26/2012	**8.9**	672 - 1119
BHSM07/SS 0-0.5	60129815007	9/26/2012	**41.5**	711 - 2285
BHSM08/SS 0-0.5	60129815008	9/26/2012	**12**	184 - 3440
BHSM08A/SS 0-0.5	60129815009	9/26/2012	**12.6**	184 - 3440
BHSM09/SS 0-0.5	60129815010	9/26/2012	**275**	442 - 1164
BHSM10/SS 0-0.5	60129815011	9/26/2012	**11.9**	912 - 1737
BHSM11/SS 0-0.5	60129815012	9/26/2012	**12.4**	1969 - 2153
BHSM12/SS 0-0.5	60129815013	9/26/2012	**83.6**	225 - 3892
BHSM13/SS 0-0.5	60129815014	9/26/2012	**32.5**	1437 - 2065

Notes:
Bold = Compound was detected
= Highlighted valued exceeds the USEPA RSL for Industrial Soil for Elemental Mercury of 43 mg/kg
[1] = Mercury vapor field screening value was obtained from surface material placed in the respective sample containers.
ID = Identification
mg/kg = milligram per kilogram
ng/m^3 = nanogram per cubic meter
USEPA RSL = United States Environmental Protection Agency Regional Screening Level

Table 4-1
Surface Material Sampling Plan
Boiler House Mercury Investigation Report
AK Steel Facility - Kansas City, Missouri

Sample Point	Sample Designator & Point	Sample Type*	Estimated Depth of Sample (ft bgs)	Total Mercury (SW846 7471)	Mercury Speciation (SOP BR-0013)
BHSM09A	SS0-0.5		0.0 - 0.5	X	X
BHSM09A	SS0-0.5A	Duplicate	0.0 - 0.5	X	X
BHSM12A	SS0-0.5		0.0 - 0.5	X	X

Notes:

* - Locations shown for QA/QC samples are preliminary and may be altered based on the order in which samples are collected, the amount of sample available, etc.

ft bgs - Feet below ground surface

SOP - Standard Operating Procedure

BR-0013 - Five-Step Selective Sequential Extraction Procedure (SEP) to Quantify Mercury Fractions in Sediments, Soils, and Mine Tailings. Brooks Rand Labs.

Table 4-2
Analytical Methods, Containers, Preservatives, and Holding Times Summary
Boiler House Mercury Investigation Report
AK Steel Facility - Kansas City, Missouri

Matrix	Analysis	Method(s)	Container Type[1]	Volume/ Mass	Preservative	Holding Time
Soil[2]	Total Mercury	SW-846 7471	One 4-ounce or 8-ounce glass jar with Teflon-Lined Lid	Fill to capacity	Ice to 4°C for Shipment Frozen for Lab Storage	1 year
	Mercury Speciation	SOP BR-0013				

°C - degrees Celsius

MS/MSD - matrix spike/matrix spike duplicate

Notes:

1 Bottle requirements were based on typical lab requirements. The lab will indicate appropriate sampling containers to meet their volume needs, and this may differ from those indicated here. Quality control samples (field duplicates, MS, and MSD) require the same containers and volume as a typical field sample unless otherwise notified by the lab.

2 Soil is to be reported on a dry-weight basis. Sufficient volume is available to perform moisture content analyses using the volume collected for the primary chemical analysis.

SOP - Standard Operating Procedure

BR-0013 - Five-Step Selective Sequential Extraction Procedure (SEP) to Quantify Mercury Fractions in Sediments, Soils, and Mine Tailings. Brooks

FIGURES

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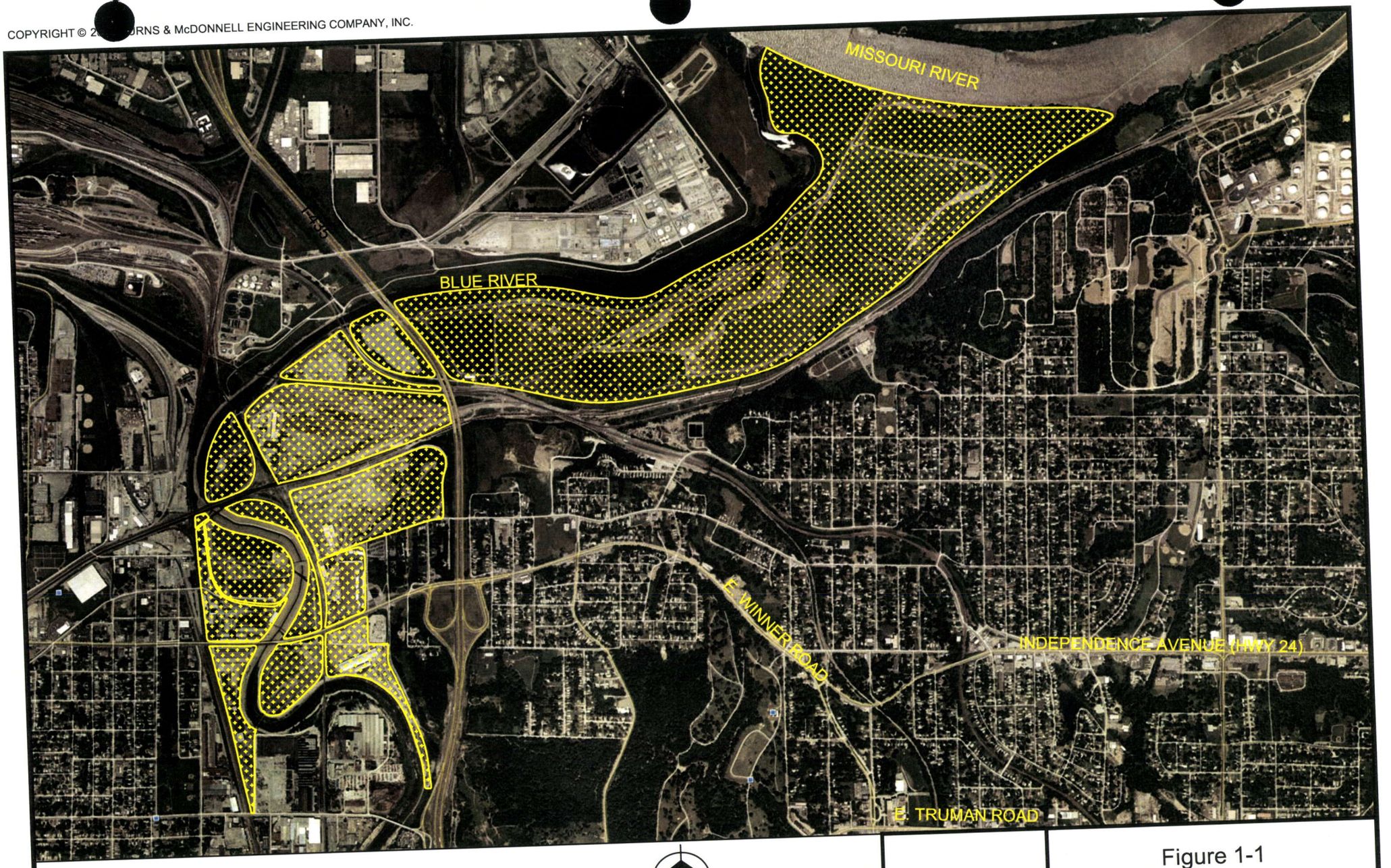


LEGEND:

AK STEEL RCRA FACILITY BOUNDARY

NORTH

0 2000' 4000'

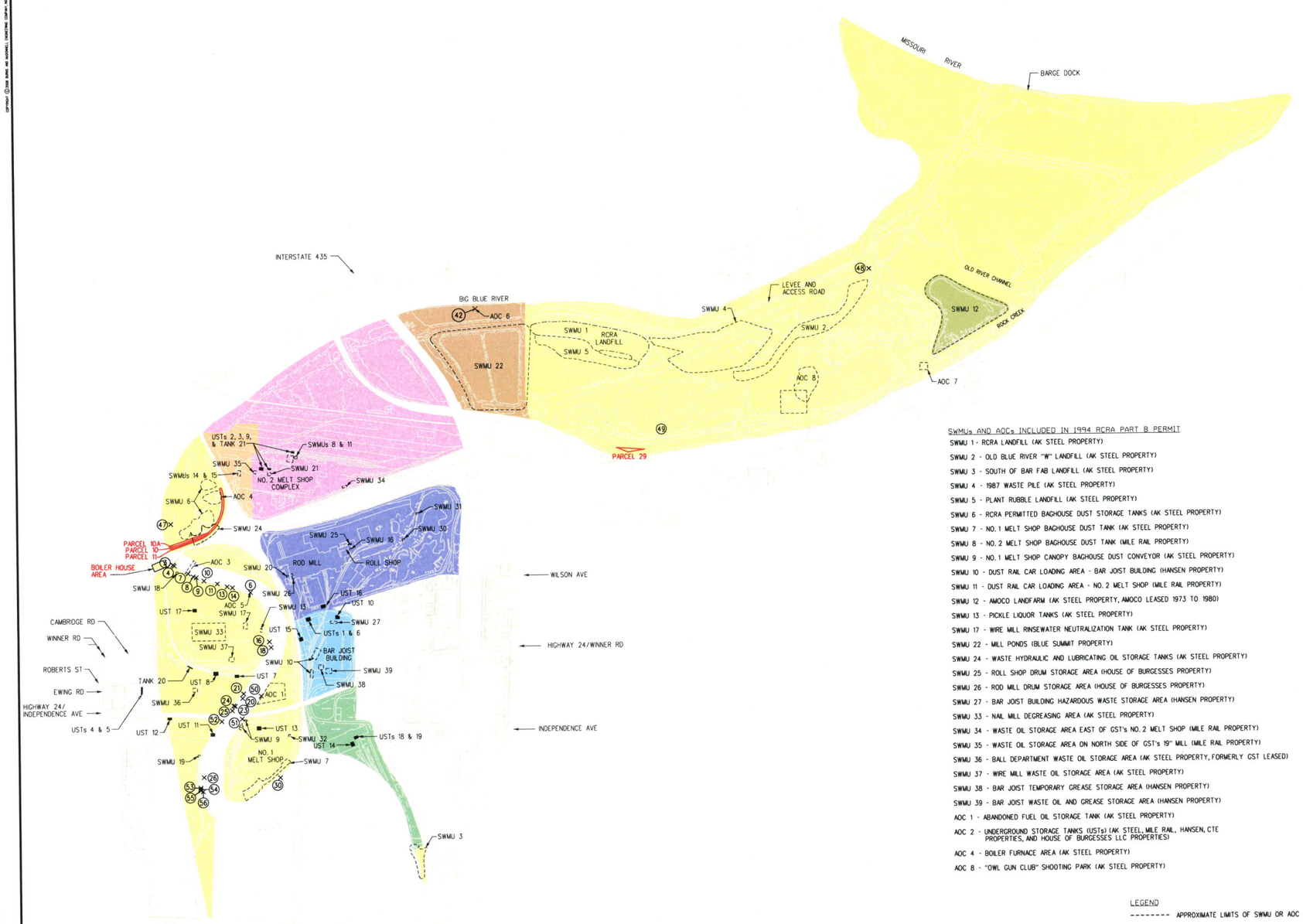
SCALE IN FEET

Burns & McDonnell SINCE 1898

Figure 1-1

FACILITY LOCATION MAP
AK STEEL
KANSAS CITY, MISSOURI

C:\DRAWING1.DWG 11-25-2008 11:06 KSCHUTTE



42 X 30

SWMUs AND AOCs INCLUDED IN 1994 RCRA PART B PERMIT

SWMU 1 - RCRA LANDFILL (AK STEEL PROPERTY)
SWMU 2 - OLD BLUE RIVER "W" LANDFILL (AK STEEL PROPERTY)
SWMU 3 - SOUTH OF BAR FAB LANDFILL (AK STEEL PROPERTY)
SWMU 4 - 1987 WASTE PILE (AK STEEL PROPERTY)
SWMU 5 - PLANT RUBBLE LANDFILL (AK STEEL PROPERTY)
SWMU 6 - RCRA PERMITTED BAGHOUSE DUST STORAGE TANKS (AK STEEL PROPERTY)
SWMU 7 - NO. 1 MELT SHOP BAGHOUSE DUST TANK (AK STEEL PROPERTY)
SWMU 8 - NO. 2 MELT SHOP BAGHOUSE DUST TANK (MILE RAIL PROPERTY)
SWMU 9 - NO. 1 MELT SHOP CANOPY BAGHOUSE DUST CONVEYOR (AK STEEL PROPERTY)
SWMU 10 - DUST RAIL CAR LOADING AREA - BAR JOIST BUILDING (HANSEN PROPERTY)
SWMU 11 - DUST RAIL CAR LOADING AREA - NO. 2 MELT SHOP (MILE RAIL PROPERTY)
SWMU 12 - AMOCO LANDFARM (AK STEEL PROPERTY, AMOCO LEASED 1973 TO 1980)
SWMU 13 - PICKLE LIQUOR TANKS (AK STEEL PROPERTY)
SWMU 17 - WIRE MILL RINSEWATER NEUTRALIZATION TANK (AK STEEL PROPERTY)
SWMU 22 - MILL PONDS (BLUE SUMMIT PROPERTY)
SWMU 24 - WASTE HYDRAULIC AND LUBRICATING OIL STORAGE TANKS (AK STEEL PROPERTY)
SWMU 25 - ROLL SHOP DRUM STORAGE AREA (HOUSE OF BURGESSES PROPERTY)
SWMU 26 - ROD MILL DRUM STORAGE AREA (HOUSE OF BURGESSES PROPERTY)
SWMU 27 - BAR JOIST BUILDING HAZARDOUS WASTE STORAGE AREA (HANSEN PROPERTY)
SWMU 33 - NAIL MILL DEGREASING AREA (AK STEEL PROPERTY)
SWMU 34 - WASTE OIL STORAGE AREA EAST OF GST's NO. 2 MELT SHOP (MILE RAIL PROPERTY)
SWMU 35 - WASTE OIL STORAGE AREA ON NORTH SIDE OF GST's 19" MILL (MILE RAIL PROPERTY)
SWMU 36 - BALL DEPARTMENT WASTE OIL STORAGE AREA (AK STEEL PROPERTY, FORMERLY GST LEASED)
SWMU 37 - WIRE MILL WASTE OIL STORAGE AREA (AK STEEL PROPERTY)
SWMU 38 - BAR JOIST TEMPORARY GREASE STORAGE AREA (HANSEN PROPERTY)
SWMU 39 - BAR JOIST WASTE OIL AND GREASE STORAGE AREA (HANSEN PROPERTY)
AOC 1 - ABANDONED FUEL OIL STORAGE TANK (AK STEEL PROPERTY)
AOC 2 - UNDERGROUND STORAGE TANKS (USTs) (AK STEEL, MILE RAIL, HANSEN, CTE PROPERTIES, AND HOUSE OF BURGESSES LLC PROPERTIES)
AOC 4 - BOILER FURNACE AREA (AK STEEL PROPERTY)
AOC 8 - "OWL GUN CLUB" SHOOTING PARK (AK STEEL PROPERTY)

SWMUs AND AOCs NOT INCLUDED IN 1994 RCRA PART B PERMIT

SWMU 14 - ETCH LAB MIXING TANK (ONESTEEL PROPERTY)
SWMU 15 - ETCH LAB HOLDING TANK (ONESTEEL PROPERTY)
SWMU 16 - ROLL SHOP ROLL CLEANING TANK (HOUSE OF BURGESSES PROPERTY)
SWMU 18 - BLOOMING MILL SCALE PIT (AK STEEL PROPERTY)
SWMU 19 - TWELVE INCH (12") MILL SCALE PIT (AK STEEL PROPERTY)
SWMU 20 - ROD MILL SCALE PIT (HOUSE OF BURGESSES PROPERTY)
SWMU 21 - NO. 2 MELT SHOP SCALE PIT (MILE RAIL PROPERTY)
SWMU 23 - SAFETY-KLEEN UNITS
SWMU 28 - OUTSIDE HAZARDOUS WASTE STORAGE AREA
SWMU 29 - MAIN SUBSTATION PCB STORAGE AREA
SWMU 30 - LONG TRACTOR SHED PCB STORAGE AREA (HOUSE OF BURGESSES PROPERTY)
SWMU 31 - SMALL TRACTOR SHED PCB STORAGE AREA (HOUSE OF BURGESSES PROPERTY)
SWMU 32 - NO. 1 MELT SHOP PCB STORAGE AREA (AK STEEL PROPERTY)
AOC 3 - ARMCO DAM/PCB EXCAVATION AREA OF BLUE RIVER (AK STEEL PROPERTY)
AOC 5 - OUTFALL NO. 006 (AK STEEL PROPERTY)
AOC 6 - OUTFALL NO. 042 (BLUE SUMMIT PROPERTY)
AOC 7 - BACKWASH AREA OF ROCK CREEK (AK STEEL PROPERTY)

LEGEND

- APPROXIMATE LIMITS OF SWMU OR AOC AREA
- BOUNDARY OF PROPERTY SOLD TO KC TERMINAL RAILROAD
- (29) OUTFALL
- LOCATION OF FORMER USTs BELONGING TO AOC 2
- AK STEEL PROPERTY
- MILE RAIL LLC PROPERTY (FORMERLY GST)
- AK STEEL PROPERTY FORMERLY LEASED TO GST
- AK STEEL PROPERTY FORMERLY LEASED TO AMOCO
- HANSEN PROPERTY DEVELOPMENT, INC. (FORMERLY GST) D.B.A. U-PICK-IT OF KANSAS CITY
- HOUSE OF BURGESSES LLC PROPERTY (FORMERLY GST) D.B.A. MIDWEST SCRAP MANAGEMENT
- CTE PROPERTIES LLC PROPERTY (FORMERLY GST) D.B.A. CUSTOM TRUCK & EQUIPMENT
- ONE STEEL GRINDING SYSTEMS LLC PROPERTY (FORMERLY GST)
- BLUE SUMMIT LLC (FORMERLY GST)

NOTES:

1. APPROXIMATE FACILITY BOUNDARY, AS DEFINED IN PERMIT, IS SHOWN. AS INDICATED, PORTIONS OF THE FACILITY ARE NO LONGER OWNED BY AK STEEL.
2. AREAS HISTORICALLY DECOMMISSIONED AND DEMOLISHED: NO. 1 MELT SHOP, ROD MILL, ROLL SHOP, AND NO. 2 MELT SHOP.
3. LOCATIONS OF SWMUS 23, 28, AND 29 ARE UNKNOWN.
4. LOCATIONS OF SWMUS AND AOCS NOT INCLUDED IN THE 1994 RCRA PART B PERMIT ARE APPROXIMATE AND NOT BASED ON SURVEY INFORMATION. THE LOCATIONS OF THESE SWMUS AND AOCS ARE BASED UPON FIGURE 6 OF THE RCRA FACILITY ASSESSMENT (TETRA TECH, 1992).

NORTH

500' 0' 500' 1000'
SCALE IN FEET

Burns & McDonnell

Figure 1-2
FACILITY MAP
AK STEEL KANSAS CITY FACILITY

NOTE:

LEGEND
LINE 1
LINE 2
LINE 3

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40 20 0 40
Feet

NORTH

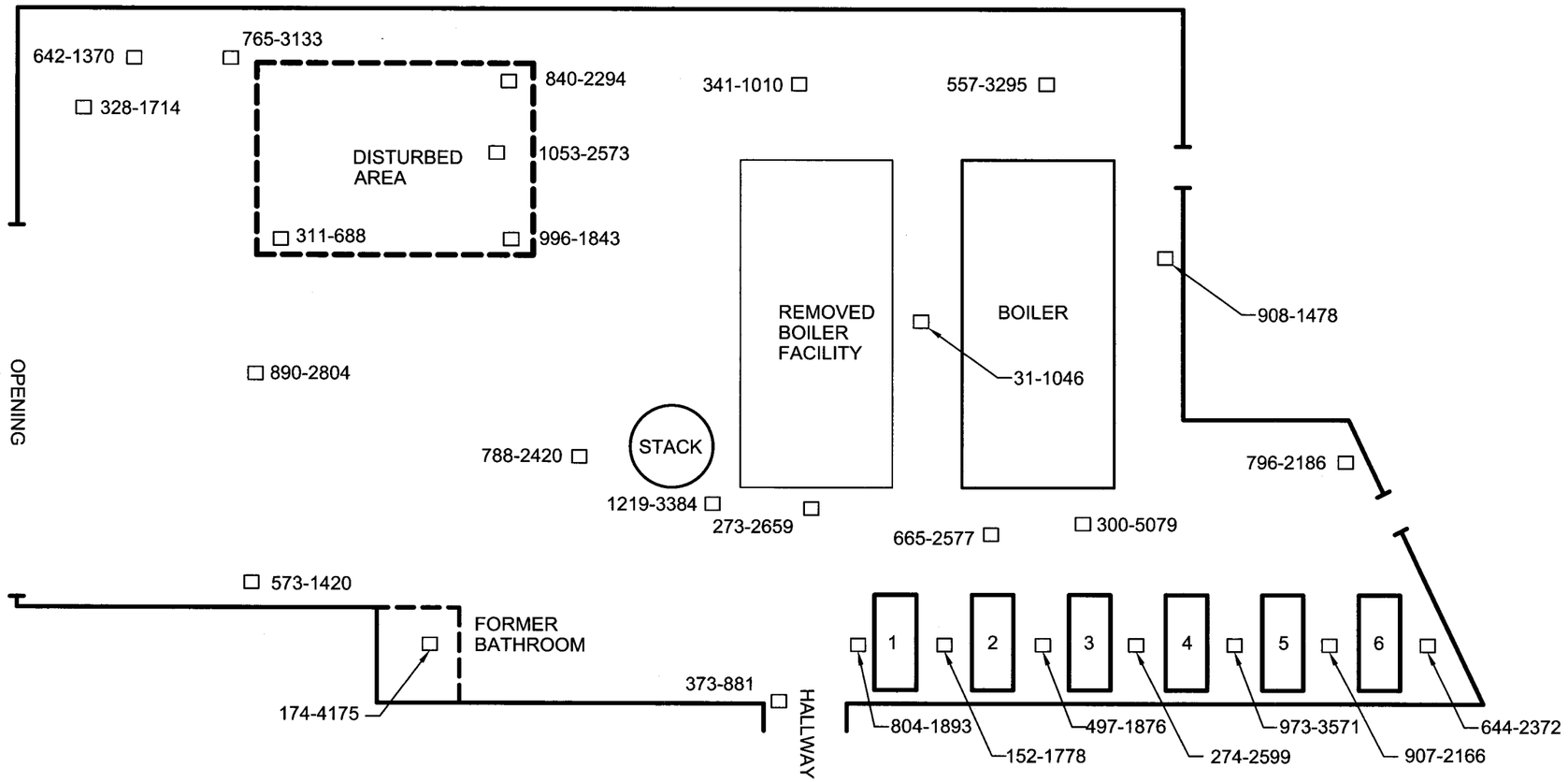
Source = NAVTEQ, 2012 / Microsoft, 2012

Burns & McDonnell
SINCE 1898

Figure 1-3
Boiler House
AK Steel Kansas City Facility
Kansas City, Missouri

Name: Figure 1-3_Boiler House_BRH

Date Saved: 8/13/2012 7:43:40 AM



N

0 10' 20'

SCALE IN FEET

NOTES:

□ APPROXIMATE LOCATION MERCURY VAPOR READINGS WERE RECORDED.

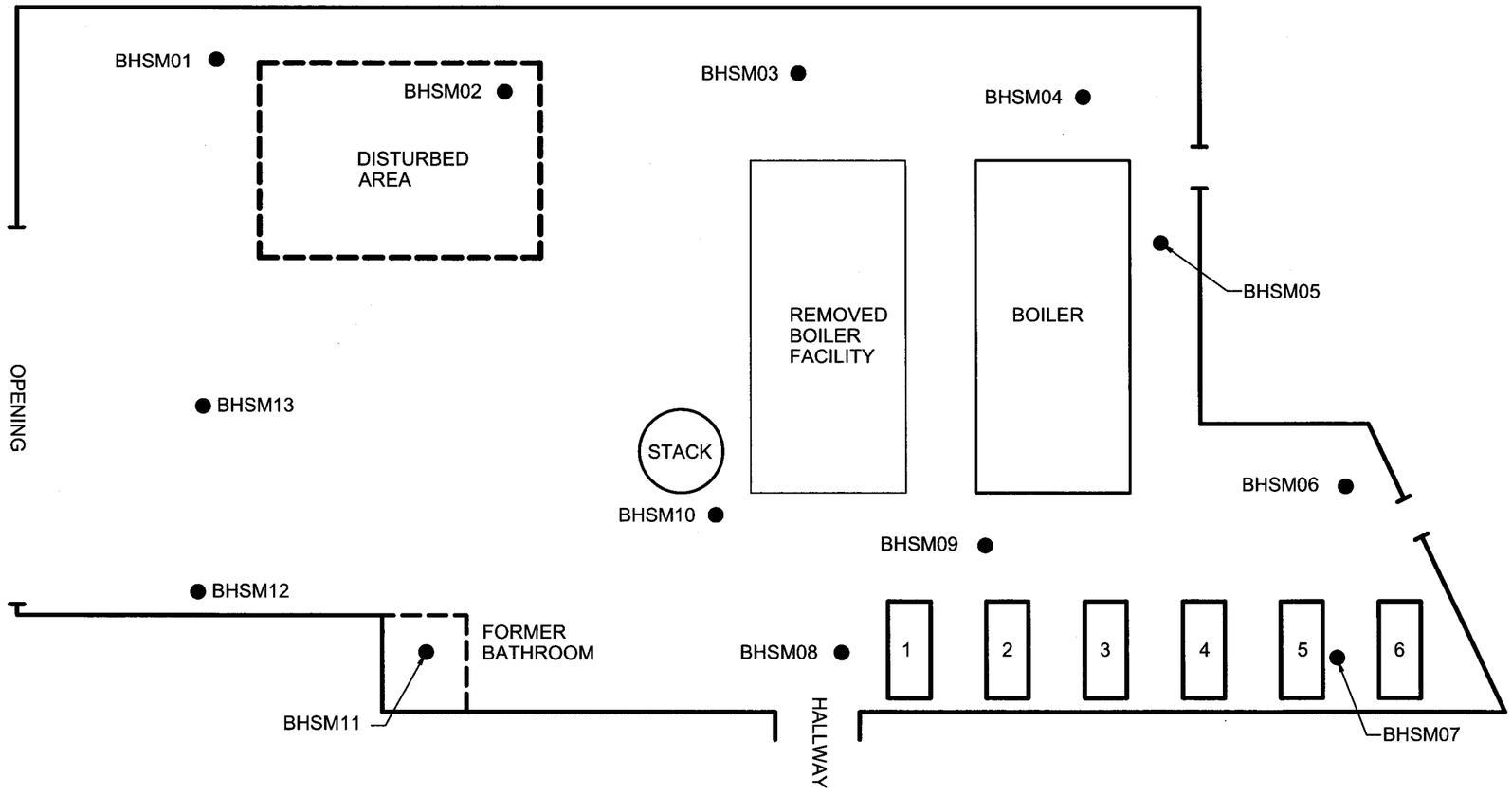
1. UNITS ARE NANOGRAMS PER CUBIC METER (ng/m ^{3})
2. FOMER EQUIPMENT MOUNT FOOTINGS ARE LOCATED ALONG THE SOUTH WALL. THESE WERE NUMBERED 1 TO 6.

Burns & McDonnell SINCE 1898

Figure 3-1
MERCURY VAPOR SCREENING LOCATIONS
AK STEEL
KANSAS CITY FACILITY

COPYRIGHT © 2012 BURNS & McDONNELL ENGINEERING COMPANY, INC.

K:\ENV\AK STEEL\SITE\60048\CAD\KC FACILITY FIGURES\AK STEEL FIGURE 3-1-MERCURY VAPOR.DWG 11/2/2012 2:10 PM TDUCKER



LEGEND:

● SURFACE MATERIAL SAMPLE LOCATION

NOTES:

1. SAMPLE LOCATIONS ARE APPROXIMATE.
2. FORMER EQUIPMENT MOUNT FOOTINGS ARE LOCATED ALONG THE SOUTH WALL. THESE WERE NUMBERED 1 TO 6.

Figure 3-2
BOILER HOUSE
SAMPLE LOCATIONS
AK STEEL
KANSAS CITY FACILITY

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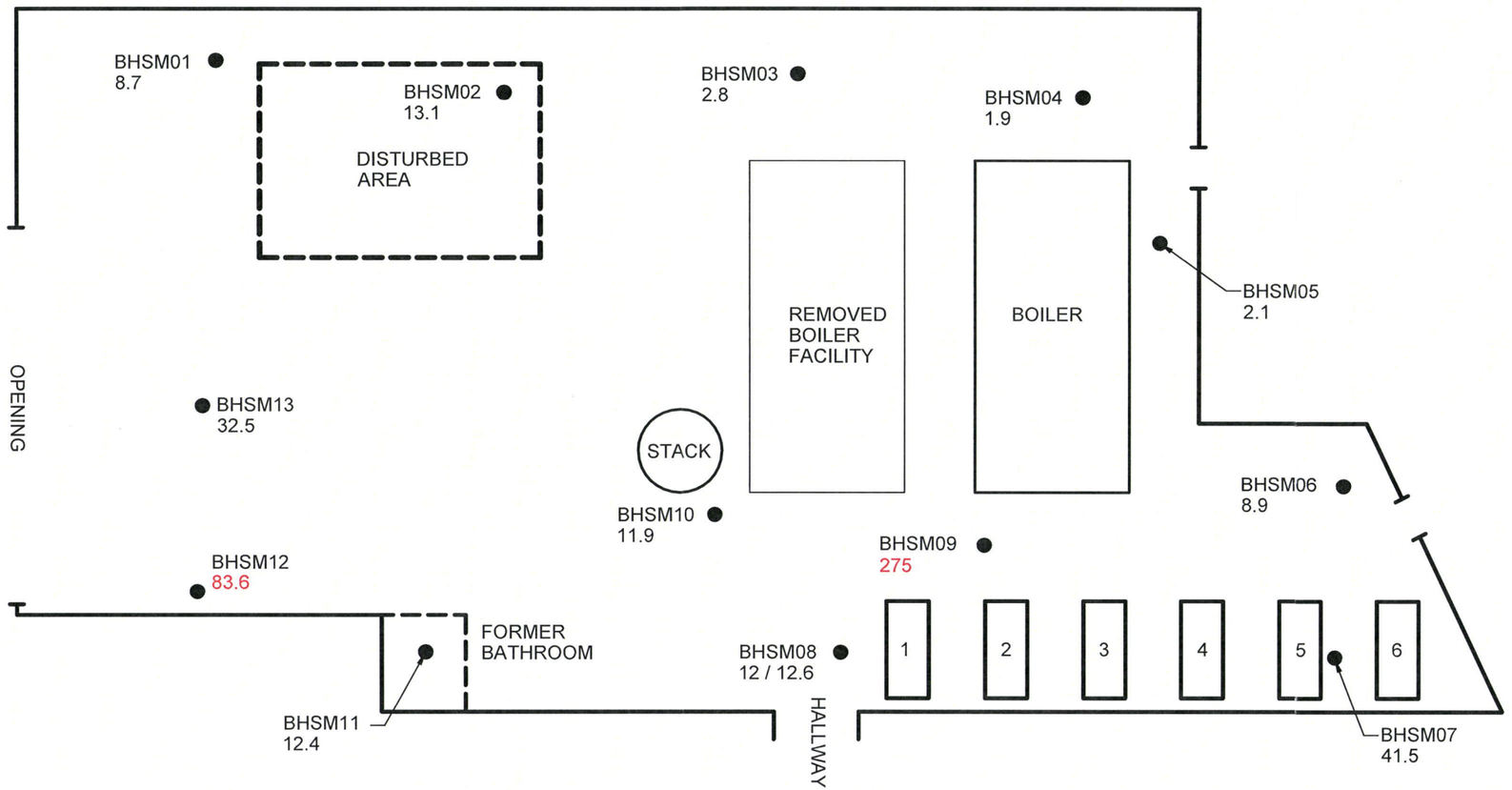
K:\ENV\AK STEEL\SITE\60048\CAD\KC FACILITY FIGURES\AKA STEEL FIGURE 3-2.DWG 10/30/2012 4:09 PM TDUCKER



LEGEND:

- SURFACE MATERIAL SAMPLE LOCATION

NOTES:

1. SAMPLE LOCATIONS ARE APPROXIMATE.
2. FORMER EQUIPMENT MOUNT FOOTINGS ARE LOCATED ALONG THE SOUTH WALL. THESE WERE NUMBERED 1 TO 6.
3. UNITS ARE MILLIGRAMS PER KILOGRAM (mg/kg).
3. VALUES EXCEEDING USEPA RSL ARE SHOWN IN RED.
4. DUPLICATE SAMPLE COLLECTED FROM LOCATION BHSM08.BOTH RESULTS ARE PRESENTED.

N

0 10' 20'

SCALE IN FEET

Burns & McDonnell SINCE 1898

Figure 3-3
BOILER HOUSE
MERCURY RESULTS
AK STEEL
KANSAS CITY FACILITY

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APPENDICES

APPENDIX A

NRC Incident Report

NATIONAL RESPONSE CENTER 1-800-424-8802
*** For Public Use ***
Information released to a third party shall comply with any
applicable federal and/or state Freedom of Information and Privacy Laws

Incident Report # 1002004

INCIDENT DESCRIPTION

*Report taken at 15:26 on 02-FEB-12
Incident Type: FIXED
Incident Cause: DUMPING
Affected Area:
The incident occurred on 01-JAN-97 at 12:00 local time.
Affected Medium: LAND BURIED INTO THE GROUND (UNDERNEATH BASEMENT FLOOR)

SUSPECTED RESPONSIBLE PARTY

Organization: GS TECHNOLOGIES
XX

Type of Organization: PRIVATE ENTERPRISE

INCIDENT LOCATION

County: JACKSON
City: KANSAS CITY State: MO

GS TECHNOLOGIES PLANT

RELEASED MATERIAL(S)

CHRIS Code: MCR Official Material Name: MERCURY
Also Known As:
Qty Released: 300 POUND(S)

DESCRIPTION OF INCIDENT

CALLER IS MAKING A REPORT INVOLVING INFORMATION THEY RECEIVED REGARDING THE RESPONSIBLE PARTY BURYING SURPLUS MERCURY INTO THE GROUND AT THE PLANT PROPERTY. CALLER WAS TOLD THE INCIDENT OCCURRED DURING THE PERIOD THE RESPONSIBLE PARTY'S COMPANY WAS GOING BANKRUPT (1990'S). CALLER WAS ALSO TOLD THE TOTAL AMOUNT OF MERCURY THAT WAS BURIED WAS BETWEEN 125 - 300 POUNDS. LIMITED LOCATION INFORMATION WAS GIVEN BUT IT WAS DESCRIBED AS ON THE RESPONSIBLE PARTY'S PLANT PROPERTY AND THEY WERE BURIED UNDER THE BOILER ROOM PLANT UNDER THE BASEMENT FLOOR.

INCIDENT DETAILS

Package: NO
Building ID:
Type of Fixed Object: OTHER
Power Generating Facility: NO
Generating Capacity:
Type of Fuel:
NPDES:
NPDES Compliance: UNKNOWN

DAMAGES

Fire Involved:	NO	Fire Extinguished: UNKNOWN		
INJURIES:	NO	Hospitalized:	Empl/Crew:	Passenger:
FATALITIES:	NO	Empl/Crew:	Passenger:	Occupant:
EVACUATIONS:	NO	Who Evacuated:	Radius/Area:	
Damages:	NO			

Closure Type	Description of Closure	Length of Closure	Direction of Closure
Air:	N		

Road: N

Major Artery: N

Waterway: N

Track: N

Passengers Transferred: NO
Environmental Impact: UNKNOWN
Media Interest: NONE Community Impact due to Material:

REMEDIAL ACTIONS

CALLER IS MAKING NOTIFICATIONS.
Release Secured: UNKNOWN
Release Rate:
Estimated Release Duration:

WEATHER

ADDITIONAL AGENCIES NOTIFIED

Federal: NONE
State/Local: NONE
State/Local On Scene:
State Agency Number:

NOTIFICATIONS BY NRC

ATLANTIC STRIKE TEAM (MAIN OFFICE)
02-FEB-12 15:42
USCG ICC (ICC ONI)
02-FEB-12 15:42
GIS RAO ST. LOUIS (COMMAND CENTER)
02-FEB-12 15:42
DOT CRISIS MANAGEMENT CENTER (MAIN OFFICE)
02-FEB-12 15:42
U.S. EPA VII (MAIN OFFICE)
02-FEB-12 15:44
FEMA REGION 7 (COORDINATION CENTER)
02-FEB-12 15:42
IA U.S. ATTORNEY'S OFFICE (INTELLIGENCE OFFICER)
02-FEB-12 15:42
MO INFORMATION ANALYSIS CENTER (COMMAND CENTER)
02-FEB-12 15:42
MO DEPT OF HEALTH AND SENIOR SVC (COMMAND CENTER)
02-FEB-12 15:42
MO OFFICE OF HOMELAND SECURITY (COMMAND CENTER)
02-FEB-12 15:42
NATIONAL INFRASTRUCTURE COORD CTR (MAIN OFFICE)
02-FEB-12 15:42
NOAA RPTS FOR MO (MAIN OFFICE)
02-FEB-12 15:42
SECTOR UPPER MISSISSIPPI RIVER (COMMAND CENTER)
02-FEB-12 15:45
DEPT HEALTH AND ENV (MAIN OFFICE)
02-FEB-12 15:42
MODNR ATTN: DUTY OFFICER (MAIN OFFICE)
02-FEB-12 15:42
DOI/OEPC DENVER (MAIN OFFICE)
02-FEB-12 15:42
USCG DISTRICT 8 (MAIN OFFICE)
02-FEB-12 15:42

ADDITIONAL INFORMATION

CALLER HAD NO ADDITIONAL COMMENTS.

*** END INCIDENT REPORT # 1002004 ***

The National Response Center is strictly an initial report taking agency and does not participate in the investigation or incident response. The NRC receives initial reporting information only and notifies Federal and State On-Scene Coordinators for response. The NRC does not verify nor does it take follow-on incident information. Verification of data and incident response is the sole responsibility of Federal/State On-Scene Coordinators. Data contained within the FOIA Web Database is initial information only. All reports provided via this server are for informational purposes only. Data to be used in legal proceedings must be obtained via written correspondence from the NRC.

APPENDIX B

1993 Plant Map with Boiler House and Ball Department Boiler Room

MILL WATER POND

MILL WATER POND

PUMP STA

SKULL DROP

COIL STORAGE

SLAG PROCESSING

TERMINAL SCRAP YARD

SCALE

NO. 2 MELT SHOP

CASTER

15" MILL

HARD PCE

SUBSTATION

HELICOPTER PAD

SCRAP MAT'L OFFICE

BLUE RIVER

MARSH

INTERSTATE HIGHWAY 435

WINNER RD

SKILES

GATE NO. 6

TULLIE

U.S. HW N°24

10" MILL ROPE DRIVE

UPPER DIRT ALLEY (STORAGE)

POTTER

WILSON RD

SMALLEY

DIRT ALLEY

CAR BOTTOM FURNACE

ROD PROCESS

OLD GOLDBERG BLDG

STORES WHSE

PARKING

MANCHESTER

GATE NO.5

BAR JOIST

ROLL STOCK

MTR RM

BILLET STOR

#2 ROD MILL

MILL BLDG

COMP STOR & SHIPPING BLDG

VIKING & YARD OFFICE

CHEM LAB

ICE HOUSE

ELECT SHOP

GATE NO.6

KCP&L SUB STATION N°53

TRUCK SCALE

TRUCK SHIPPING OFFICE

BAR FABRICATING BLDG.

RR TRACK

SOLD

ALL BRIGHT GALVANIZING

VENDO

SOLD

NO.1 MELT SHOP

BIG

RIVER

BALL SHIPPING

OLD WIRE MILL AREA

Ball Department Boiler Room approximate location. Armco/AK Steel property formerly leased to GST.

LOCO SHOP

BALL DEPT. #17

BALL DEPT. #18

BALL DEPT. #19

BALL DEPT. #20

BALL DEPT. #21

MERCHANT BAR WHSE N°9 BLDG

SPIKE DEPT.

GR. ROD

HOT BOLT & NUT

MILL WHSE #16 GALV

MACHINE SHOP

MILLWRIGHT SHOP

STORE ROOM

SCALE HOUSE

BOILER ROOM

CONDITIONING

PARKING

AMBULANCE

GENERAL OFFICES

LOCKER ROOM

GATE 1

ASSET MGMT.

SHAW & SONS

GARAGE

SHOP

CREDIT UNION

US 24

12" MILL SHIPPING N°10 BLDG

WAREHOUSE BLDG N°11 BLDG

BILLET STORAGE BLDG

BOLT & FORGED PRODUCTS SOLD

SOLD

BLDG C

7TH ST

8TH ST

9TH ST

10TH ST

12TH ST

CAMBRIDGE

EWING

WILSON

WINNER

ROBERTS

Former Boiler House Building. Armco/AK Steel Property

200 0 200 400
SCALE IN FEET

200 0 200 400
SCALE IN FEET

REFERENCE DRAWINGS

REVISIONS NO	LOC	BY	DATE	# FILM	DESCRIPTION
	SAS		6/4/93		UPDATE DWG
	SAS		4/13/93		UPDATE DWG
	SAS		2/26/93		UPDATE DWG

ARMCO WORLDWIDE GRINDING SYSTEMS
KANSAS CITY WORKS
7000 Roberts Street
Kansas City, Mo. 64125

PLANT MAP

DWN. BY	DATE 2/26/93
APPROVED	SCALE 1"=200'
DWG. NO. 26002	REV. 3

APPENDIX C

QA/QC Review of Analytical Data

Date: October 16, 2012

To: Sharon Shelton

From: Ryann Odor

Re: Quality Assurance/Quality Control (QA/QC) Review of Analytical Data
AK Steel, Kansas City, MO
Project No: 69635

Surface Soil samples were collected as part of a mercury investigation in the Boiler House at AK Steel in Kansas City, MO (Site). Soil samples were collected on September 26, 2012. All samples were submitted to Pace Analytical Services in Lenexa, Kansas (Pace) for analysis of mercury by the analytical method EPA 7471 and percent moisture by the analytical method ASTM D2974.

The QA/AC results in association with the samples collected were examined for any method specific requirements. Data qualifiers, when appropriate, were added to the data as recommended in United States Environmental Protection Agency's (USEPA's) *National Functional Guidelines for Inorganic Superfund Data Review* (NFGO, 2010) and *National Functional Guidelines for Superfund Organic Methods Data Review* (NFGO, 2008). The QA/QC review results are discussed below.

1. Chain-of-Custody (COC) – The relinquished and received signatures, times, and dates on the COC were present and properly signed.

2. Requested Analysis Completed – All analysis were completed as requested.

3. Holding Times – All samples were analyzed within the recommended method holding times.

4. Sample Preservation – The sample coolers were received within the 4 degrees Celsius (°C) ± 2 °C sample preservation temperature range. Upon arrival at the lab, all samples were logged-in, placed in the laboratory cooler, and kept at temperatures between 2 and 6 °C.

5. Laboratory Method Blanks – All method blanks were non-detect for target analytes.

6. Laboratory Control Sample (LCS) – The LCS contains a matrix similar to that of the sample that has been spiked with known concentrations of target analytes. The LCS is prepared and analyzed by the same method as the samples. As a measure of analytical accuracy, the results of the LCS are compared against the known analyte concentrations in the spike to determine REC. The purpose of the LCS is to determine the performance of the laboratory with respect to analyte recovery, independent of field sample matrix interference.

   The LCS REC was within QC limits.

7. Matrix Spike/Matrix Spike Duplicates (MS/MSD) – MS/MSDs are typically run for organic and inorganic analyses. A sample is split into three portions (original, MS, and MSD), and a known amount of a target analyte is added (spiked) to two portions (MS and MSD) of the sample. The results of these two portions are compared with each other for reproducibility using the relative

percent difference (RPD). They are also compared against the unspiked portion of the sample for REC of the spike.

The MS/MSD analysis for QC Batch MERP/6666 was performed on sample BHSM01/SS 0-0.5 (60129815001) and had RECs that were outside of QC limits for mercury. The lab noted that the MS/MSD recovery was not evaluated against control limits due to high sample dilution and the spiked amounts were less than one-fourth the parent sample concentrations. Therefore, conclusions could not be drawn and no qualifiers were added based on this analysis.

8. Field Duplicate – Field duplicate results provide information on the ability to reproduce field results and account for error introduced from handling, shipping, storage, preparation, and analysis of field samples. There are no specific USEPA criteria for qualifying data from field duplicate results. Depending upon the sample concentration, one of the following criteria based upon NFGI is applicable:

   - Is the compound detected in both portions?
   - If the sample concentrations are greater than 5 times the detection limit, then the maximum allowable RPD is 35% percent for soil samples.
   - If the sample concentrations are less than 5 times the detection limit, then a sensitivity test is applied. For the sensitivity test, the sample concentrations must agree within plus or minus (±) two times the lower detection limit for soil samples.

   The following field duplicate pair was gathered: BHSM08/SS 0-0.5 (60129815008) and BHSM08A/SS 0-0.5 (60129815009). The mercury results were adequately replicated.

9. Laboratory Duplicate Results – Laboratory duplicate analyses were performed for percent moisture. The laboratory duplicate results met the QC criteria.

10. Detection and Quantitation Limits – Table 1 presents the analyses that required a dilution to bring concentrations of target analytes within the calibration range and/or to account for matrix interference(s). These dilutions resulted in an elevated reporting limit. All samples required a dilution of 5:1 or greater.

11. Conclusion – The data were reviewed for achievement of any method-specified QA/QC criteria. No data qualifiers were added and no data were rejected (R) as a result of this review. The data are valid for use in reporting the results of this investigation.

Attachment

Table 1 – Dilution Factors

Table 1
Dilution Factors
Boiler House Mercury Investigation
AK Steel, Kansas City, Missouri

Sample Identification	Laboratory Number	Parameter	Dilution Factor
BHSM04/SS 0-0.5	60129815004	Mercury	5
BHSM05/SS 0-0.5	60129815005		
BHSM03/SS 0-0.5	60129815003	Mercury	10
BHSM01/SS 0-0.5	60129815001	Mercury	20
BHSM02/SS 0-0.5	60129815002		
BHSM06/SS 0-0.5	60129815006		
BHSM08/SS 0-0.5	60129815008		
BHSM08A/SS 0-0.5	60129815009		
BHSM011/SS 0-0.5	60129815012		
BHSM07/SS 0-0.5	60129815007	Mercury	50
BHSM010/SS 0-0.5	60129815011		
BHSM013/SS 0-0.5	60129815014		
BHSM012/SS 0-0.5	60129815013	Mercury	250
BHSM09/SS 0-0.5	60129815010	Mercury	500

APPENDIX D

September 26, 2012 Field Notes
Surface Material Sampling

69635

TUESDAY, JULY 24, 2012

TASK: BOILER HOUSE DIMENSION MEASUREMENTS

WEATHER: CLEAR, UPPER 90's.

PERSONNEL: DANIEL EARHART

0745 LEAVE KC OFFICE

0820 ARRIVE AT AK STEEL. FRONT GATE ALREADY OPEN.

0825 BEGIN COLLECTING MEASUREMENTS OF BOILER HOUSE.

0930 FINISH COLLECTING BOILER HOUSE MEASUREMENTS.

1000 ARRIVE BACK AT KC OFFICE.

7-24-12

69635

WEDNESDAY SEPT. 26, 2012

TASK: SOIL SAMPLING AT BOILER HOUSE

PERSONNEL: DANIEL EARHART, BRIAN HOYE, SHARON SHELTON, BRUCE

WEATHER: 60s, LIGHT RAIN, NO WIND

0850 - ARRIVE AT THE BOILER HOUSE. DON PPE (BOOT COVERS, GLOVES, EYE PROTECTION & HARD HATS.)

0855 - START UP Hg METER. BACKGROUND = 23-48

0905 - BEGIN Hg READINGS

LOCATION — READING

LOCATIONS AND READINGS RECORDED ON FIGURE TO AID IN LOCATING SAMPLING LOCATIONS.

0940 - RETURN TO TRUCK WITH RESULTS, USEPA (BRUCE MORRISON) POINTS OUT 13 LOCATIONS TO BE SAMPLED. REQUESTS THAT THE Hg METER READINGS BE CONFIRMED WHEN SAMPLING.

0955 - COLLECT BHSM01/SS0-0.5 FOR Hg. METER = 922 - 2241. TAKE READING FROM HOMOGONIZED SAMPLE = 1198 - 1224

1010 - COLLECT BHSM02/SS0-0.5 FOR Hg. METER READING @ GROUND = 2316 - 4757 METER READIN OF SAMPLE = 1997 - 2462

16

S SHELTON
B. HOVE
D. EARHART

9/26/2012 69635

1015 - COLLECT BHSM003/SSO-0.5 FOR HG.
METER READING AT THE LOCATION = 691 - 1910
METER READING OFF THE SAMPLE = 1249 - 2390
1020 - COLLECT BHSM04/SSO-0.5 FOR Hg.
METER READING AT THE LOC. = 938 - 1570
METER READING OFF THE SAMPLE = 955 - 1562
1025 - COLLECT BHSM05/SSO-0.5 FOR Hg.
METER READING AT THE LOC. = 1282 - 1771
METER READING OFF THE SAMPLE = 641 - 2020
1035 - COLLECT BHSM06/SSO-0.5 ~~+ BHSM06A/SSO-0.5~~ (BH) FOR Hg. METER READING AT THE LOCATION = 1167 - 2365
Hg METER READING OFF THE SAMPLE = 672 - 1119.
1040 - COLLECT BHSM07/SSO-0.5 FOR HG.
METER READING @ THE SAMPLE LOCATION = 1208 - 2004
METER READING OFF THE SAMPLE VOLUME = 711 - 2285
1045 - COLLECT BHSM08/SSO-0.5 + BHSM08A/SSO-0.5 FOR Hg.
METER READING AT THE LOCATION = 1069 - 284
METER READING OFF THE SAMPLE = 184 - 3440
1050 - COLLECT BHSM09/SSO-0.5 FOR Hg.
METER READING AT THE LOCATION = 1676 - 2099
METER READING OFF THE SAMPLE - 442 - 1164.
1055 - COLLECT BHSM10/SSO-0.5 + BHSM10MS/MSD/SSO-0.5 FOR Hg. METER READING AT THE LOCATION = 1308 - 1765. METER READING OFF THE SAMPLE - 917 - 1737

S. SHELTON
B. HOVE 17
D. EARHART

9/26/2012 69635

1100 - COLLECT BHSM11/SSO-0.5 FOR Hg.
METER READING AT LOCATION = 1007 - 1804
METER READING OFF SAMPLE = 1969 - 2153
1105 - COLLECT BHSM12/SSO-0.5 FOR Hg.
METER READING AT LOCATION = 680 - 1556
METER READING OFF SAMPLE = 225 - 3892
1110 - COLLECT BHSM13/SSO-0.5 FOR Hg.
METER READING AT LOCATION = 755 - 1132
METER READING OFF SAMPLE = 1437 - 2065
1118 - TAKE BACK GROUND READING = 13 - 47.
1120 MEASURE OFF SAMPLE LOCATIONS.
1125 - EPA + S. SHELTON OFFSITE
1140 - FILL OUT CHAIN.
1200 - OFFSITE. LOCK GATE WHEN LEAVING.
1350 DROP OFF SAMPLES AT PACE ANALYTICAL.

9-26-12

APPENDIX E

Analytical Laboratory Report

Pace Analytical®
www.pacelabs.com

Pace Analytical Services, Inc.
9608 Loiret Blvd.
Lenexa, KS 66219
(913)599-5665

October 08, 2012

SHARON SHELTON
BURNS & MCDONNELL
9400 WARD PARKWAY
Kansas City, MO 64131

RE: Project: Boiler House
Pace Project No.: 60129815

Dear SHARON SHELTON:
Enclosed are the analytical results for sample(s) received by the laboratory on September 26, 2012. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Angie Brown

Angie.Brown@pacelabs.com
Project Manager

Enclosures

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Pace Analytical Services, Inc.
9608 Loiret Blvd.
Lenexa, KS 66219
(913)599-5665

CERTIFICATIONS

Project: Boiler House
Pace Project No.: 60129815

Kansas Certification IDs
9608 Loiret Boulevard, Lenexa, KS 66219
A2LA Certification #: 2456.01
Arkansas Certification #: 12-019-0
Illinois Certification #: 002885
Iowa Certification #: 118
Kansas/NELAP Certification #: E-10116
Louisiana Certification #: 03055
Nevada Certification #: KS000212008A
Oklahoma Certification #: 9205/9935
Texas Certification #: T104704407-12-3
Utah Certification #: KS000212012-2

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www.pacelabs.com

Pace Analytical Services, Inc.
9608 Loiret Blvd.
Lenexa, KS 66219
(913)599-5665

SAMPLE SUMMARY

Project: Boiler House
Pace Project No.: 60129815

Lab ID	Sample ID	Matrix	Date Collected	Date Received
60129815001	BHSM01/SS 0-0.5	Solid	09/26/12 09:55	09/26/12 13:55
60129815002	BHSM02/SS 0-0.5	Solid	09/26/12 10:10	09/26/12 13:55
60129815003	BHSM03/SS 0-0.5	Solid	09/26/12 10:15	09/26/12 13:55
60129815004	BHSM04/SS 0-0.5	Solid	09/26/12 10:20	09/26/12 13:55
60129815005	BHSM05/SS 0-0.5	Solid	09/26/12 10:25	09/26/12 13:55
60129815006	BHSM06/SS 0-0.5	Solid	09/26/12 10:35	09/26/12 13:55
60129815007	BHSM07/SS 0-0.5	Solid	09/26/12 10:40	09/26/12 13:55
60129815008	BHSM08/SS 0-0.5	Solid	09/26/12 10:45	09/26/12 13:55
60129815009	BHSM08A/SS 0-0.5	Solid	09/26/12 08:00	09/26/12 13:55
60129815010	BHSM09/SS 0-0.5	Solid	09/26/12 10:50	09/26/12 13:55
60129815011	BHSM10/SS 0-0.5	Solid	09/26/12 10:55	09/26/12 13:55
60129815012	BHSM11/SS 0-0.5	Solid	09/26/12 11:00	09/26/12 13:55
60129815013	BHSM12/SS 0-0.5	Solid	09/26/12 11:05	09/26/12 13:55
60129815014	BHSM13/SS 0-0.5	Solid	09/26/12 11:10	09/26/12 13:55

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SAMPLE ANALYTE COUNT

Project: Boiler House
Pace Project No.: 60129815

Lab ID	Sample ID	Method	Analysts	Analytes Reported
60129815001	BHSM01/SS 0-0.5	EPA 7471	TDS	1
		ASTM D2974	TMD	1
60129815002	BHSM02/SS 0-0.5	EPA 7471	TDS	1
		ASTM D2974	TMD	1
60129815003	BHSM03/SS 0-0.5	EPA 7471	TDS	1
		ASTM D2974	TMD	1
60129815004	BHSM04/SS 0-0.5	EPA 7471	TDS	1
		ASTM D2974	TMD	1
60129815005	BHSM05/SS 0-0.5	EPA 7471	TDS	1
		ASTM D2974	TMD	1
60129815006	BHSM06/SS 0-0.5	EPA 7471	TDS	1
		ASTM D2974	TMD	1
60129815007	BHSM07/SS 0-0.5	EPA 7471	TDS	1
		ASTM D2974	TMD	1
60129815008	BHSM08/SS 0-0.5	EPA 7471	TDS	1
		ASTM D2974	TMD	1
60129815009	BHSM08A/SS 0-0.5	EPA 7471	TDS	1
		ASTM D2974	TMD	1
60129815010	BHSM09/SS 0-0.5	EPA 7471	TDS	1
		ASTM D2974	TMD	1
60129815011	BHSM10/SS 0-0.5	EPA 7471	TDS	1
		ASTM D2974	TMD	1
60129815012	BHSM11/SS 0-0.5	EPA 7471	TDS	1
		ASTM D2974	TMD	1
60129815013	BHSM12/SS 0-0.5	EPA 7471	TDS	1
		ASTM D2974	TMD	1
60129815014	BHSM13/SS 0-0.5	EPA 7471	TDS	1
		ASTM D2974	TMD	1

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ANALYTICAL RESULTS

Project: Boiler House
Pace Project No.: 60129815

Sample: BHSM01/SS 0-0.5 **Lab ID: 60129815001** Collected: 09/26/12 09:55 Received: 09/26/12 13:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
7471 Mercury	Analytical Method: EPA 7471 Preparation Method: EPA 7471							
Mercury	**8.7**	mg/kg	0.82	20	10/03/12 10:15	10/04/12 09:53	7439-97-6	
Percent Moisture	Analytical Method: ASTM D2974							
Percent Moisture	**3.8**	%	0.50	1		10/02/12 00:00		

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ANALYTICAL RESULTS

Project: Boiler House
Pace Project No.: 60129815

Sample: BHSM02/SS 0-0.5 **Lab ID: 60129815002** Collected: 09/26/12 10:10 Received: 09/26/12 13:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
7471 Mercury	Analytical Method: EPA 7471 Preparation Method: EPA 7471							
Mercury	**13.1**	mg/kg	0.85	20	10/03/12 10:15	10/04/12 09:55	7439-97-6	
Percent Moisture	Analytical Method: ASTM D2974							
Percent Moisture	**9.8**	%	0.50	1		10/02/12 00:00		

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ANALYTICAL RESULTS

Project: Boiler House
Pace Project No.: 60129815

Sample: BHSM03/SS 0-0.5 **Lab ID: 60129815003** Collected: 09/26/12 10:15 Received: 09/26/12 13:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
7471 Mercury	Analytical Method: EPA 7471 Preparation Method: EPA 7471							
Mercury	**2.8**	mg/kg	0.48	10	10/03/12 10:15	10/04/12 09:58	7439-97-6	
Percent Moisture	Analytical Method: ASTM D2974							
Percent Moisture	**4.9**	%	0.50	1		10/02/12 00:00		

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ANALYTICAL RESULTS

Project: Boiler House
Pace Project No.: 60129815

Sample: BHSM04/SS 0-0.5 **Lab ID: 60129815004** Collected: 09/26/12 10:20 Received: 09/26/12 13:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
7471 Mercury	Analytical Method: EPA 7471 Preparation Method: EPA 7471							
Mercury	**1.9**	mg/kg	0.29	5	10/03/12 10:15	10/04/12 10:00	7439-97-6	
Percent Moisture	Analytical Method: ASTM D2974							
Percent Moisture	**14.3**	%	0.50	1		10/02/12 00:00		

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ANALYTICAL RESULTS

Project: Boiler House
Pace Project No.: 60129815

Sample: BHSM05/SS 0-0.5 **Lab ID: 60129815005** Collected: 09/26/12 10:25 Received: 09/26/12 13:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
7471 Mercury	Analytical Method: EPA 7471 Preparation Method: EPA 7471							
Mercury	**2.1**	mg/kg	0.18	5	10/03/12 10:15	10/04/12 10:02	7439-97-6	
Percent Moisture	Analytical Method: ASTM D2974							
Percent Moisture	**4.7**	%	0.50	1		10/02/12 00:00		

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ANALYTICAL RESULTS

Project: · Boiler House
Pace Project No.: 60129815

Sample: BHSM06/SS 0-0.5 **Lab ID: 60129815006** Collected: 09/26/12 10:35 Received: 09/26/12 13:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
7471 Mercury	Analytical Method: EPA 7471 Preparation Method: EPA 7471							
Mercury	**8.9**	mg/kg	0.94	20	10/03/12 10:15	10/04/12 10:04	7439-97-6	
Percent Moisture	Analytical Method: ASTM D2974							
Percent Moisture	**3.7**	%	0.50	1		10/02/12 00:00		

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ANALYTICAL RESULTS

Project: Boiler House
Pace Project No.: 60129815

Sample: BHSM07/SS 0-0.5 **Lab ID: 60129815007** Collected: 09/26/12 10:40 Received: 09/26/12 13:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
7471 Mercury	Analytical Method: EPA 7471 Preparation Method: EPA 7471							
Mercury	**41.5**	mg/kg	2.1	50	10/03/12 10:15	10/04/12 10:06	7439-97-6	
Percent Moisture	Analytical Method: ASTM D2974							
Percent Moisture	**2.8**	%	0.50	1		10/02/12 00:00		

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ANALYTICAL RESULTS

Project: Boiler House
Pace Project No.: 60129815

Sample: BHSM08/SS 0-0.5 **Lab ID: 60129815008** Collected: 09/26/12 10:45 Received: 09/26/12 13:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
7471 Mercury	Analytical Method: EPA 7471 Preparation Method: EPA 7471							
Mercury	**12.0**	mg/kg	1.1	20	10/03/12 10:15	10/04/12 10:13	7439-97-6	
Percent Moisture	Analytical Method: ASTM D2974							
Percent Moisture	**21.3**	%	0.50	1		10/02/12 00:00		

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ANALYTICAL RESULTS

Project: Boiler House
Pace Project No.: 60129815

Sample: BHSM08A/SS 0-0.5 **Lab ID: 60129815009** Collected: 09/26/12 08:00 Received: 09/26/12 13:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
7471 Mercury	Analytical Method: EPA 7471 Preparation Method: EPA 7471							
Mercury	**12.6**	mg/kg	1.0	20	10/03/12 10:15	10/04/12 10:15	7439-97-6	
Percent Moisture	Analytical Method: ASTM D2974							
Percent Moisture	**20.1**	%	0.50	1		10/02/12 00:00		

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ANALYTICAL RESULTS

Project: Boiler House
Pace Project No.: 60129815

Sample: BHSM09/SS 0-0.5 **Lab ID: 60129815010** Collected: 09/26/12 10:50 Received: 09/26/12 13:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
7471 Mercury	Analytical Method: EPA 7471 Preparation Method: EPA 7471							
Mercury	**275**	mg/kg	22.8	500	10/03/12 10:15	10/04/12 10:42	7439-97-6	
Percent Moisture	Analytical Method: ASTM D2974							
Percent Moisture	**3.1**	%	0.50	1		10/02/12 00:00		

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ANALYTICAL RESULTS

Project: Boiler House
Pace Project No.: 60129815

Sample: BHSM10/SS 0-0.5 **Lab ID: 60129815011** Collected: 09/26/12 10:55 Received: 09/26/12 13:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
7471 Mercury	Analytical Method: EPA 7471 Preparation Method: EPA 7471							
Mercury	**11.9**	mg/kg	1.8	50	10/03/12 10:15	10/04/12 10:20	7439-97-6	M6
Percent Moisture	Analytical Method: ASTM D2974							
Percent Moisture	**4.0**	%	0.50	1		10/02/12 00:00		

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ANALYTICAL RESULTS

Project: Boiler House
Pace Project No.: 60129815

Sample: BHSM11/SS 0-0.5 **Lab ID: 60129815012** Collected: 09/26/12 11:00 Received: 09/26/12 13:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
7471 Mercury	Analytical Method: EPA 7471 Preparation Method: EPA 7471							
Mercury	**12.4**	mg/kg	1.1	20	10/03/12 10:15	10/04/12 10:26	7439-97-6	
Percent Moisture	Analytical Method: ASTM D2974							
Percent Moisture	**35.3**	%	0.50	1		10/02/12 00:00		

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ANALYTICAL RESULTS

Project: Boiler House
Pace Project No.: 60129815

Sample: BHSM12/SS 0-0.5 **Lab ID: 60129815013** Collected: 09/26/12 11:05 Received: 09/26/12 13:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
7471 Mercury	Analytical Method: EPA 7471 Preparation Method: EPA 7471							
Mercury	**83.6**	mg/kg	11.9	250	10/03/12 10:15	10/04/12 10:44	7439-97-6	
Percent Moisture	Analytical Method: ASTM D2974							
Percent Moisture	**7.4**	%	0.50	1		10/02/12 00:00		

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ANALYTICAL RESULTS

Project: Boiler House
Pace Project No.: 60129815

Sample: BHSM13/SS 0-0.5 **Lab ID: 60129815014** Collected: 09/26/12 11:10 Received: 09/26/12 13:55 Matrix: Solid

Results reported on a "dry-weight" basis

Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
7471 Mercury	Analytical Method: EPA 7471 Preparation Method: EPA 7471							
Mercury	**32.5**	mg/kg	1.9	50	10/03/12 10:15	10/04/12 10:31	7439-97-6	
Percent Moisture	Analytical Method: ASTM D2974							
Percent Moisture	**2.5**	%	0.50	1		10/02/12 00:00		

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QUALITY CONTROL DATA

Project: Boiler House
Pace Project No.: 60129815

QC Batch: MERP/6666
QC Batch Method: EPA 7471
Analysis Method: EPA 7471
Analysis Description: 7471 Mercury
Associated Lab Samples: 60129815001, 60129815002, 60129815003, 60129815004, 60129815005, 60129815006, 60129815007, 60129815008, 60129815009, 60129815010, 60129815011, 60129815012, 60129815013, 60129815014

METHOD BLANK: 1069173
Matrix: Solid
Associated Lab Samples: 60129815001, 60129815002, 60129815003, 60129815004, 60129815005, 60129815006, 60129815007, 60129815008, 60129815009, 60129815010, 60129815011, 60129815012, 60129815013, 60129815014

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Mercury	mg/kg	ND	0.050	10/04/12 09:49	

LABORATORY CONTROL SAMPLE: 1069174

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Mercury	mg/kg	.5	0.51	102	80-120	

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 1069175 1069176

Parameter	Units	60129815011 Result	MS Spike Conc.	MSD Spike Conc.	MS Result	MSD Result	MS % Rec	MSD % Rec	% Rec Limits	RPD	Max RPD	Qual
Mercury	mg/kg	11.9	.38	.38	12.7	11.2	222	-168	75-125	12	20	M6

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QUALITY CONTROL DATA

Project: Boiler House
Pace Project No.: 60129815

QC Batch: PMST/7789 Analysis Method: ASTM D2974
QC Batch Method: ASTM D2974 Analysis Description: Dry Weight/Percent Moisture
Associated Lab Samples: 60129815001, 60129815002, 60129815003, 60129815004, 60129815005, 60129815006, 60129815007, 60129815008, 60129815009, 60129815010, 60129815011, 60129815012, 60129815013, 60129815014

METHOD BLANK: 1071856 Matrix: Solid
Associated Lab Samples: 60129815001, 60129815002, 60129815003, 60129815004, 60129815005, 60129815006, 60129815007, 60129815008, 60129815009, 60129815010, 60129815011, 60129815012, 60129815013, 60129815014

Parameter	Units	Blank Result	Reporting Limit	Analyzed	Qualifiers
Percent Moisture	%	ND	0.50	10/02/12 00:00	

SAMPLE DUPLICATE: 1071857

Parameter	Units	60129815011 Result	Dup Result	RPD	Max RPD	Qualifiers
Percent Moisture	%	4.0	4.0	1	20	

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QUALIFIERS

Project: Boiler House
Pace Project No.: 60129815

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to changes in sample preparation, dilution of the sample aliquot, or moisture content.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PRL - Pace Reporting Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine (8270 listed analyte) decomposes to Azobenzene.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

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TNI - The NELAC Institute.

ANALYTE QUALIFIERS

M6 Matrix spike and Matrix spike duplicate recovery not evaluated against control limits due to sample dilution.

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QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: Boiler House
Pace Project No.: 60129815

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
60129815001	BHSM01/SS 0-0.5	EPA 7471	MERP/6666	EPA 7471	MERC/6640
60129815002	BHSM02/SS 0-0.5	EPA 7471	MERP/6666	EPA 7471	MERC/6640
60129815003	BHSM03/SS 0-0.5	EPA 7471	MERP/6666	EPA 7471	MERC/6640
60129815004	BHSM04/SS 0-0.5	EPA 7471	MERP/6666	EPA 7471	MERC/6640
60129815005	BHSM05/SS 0-0.5	EPA 7471	MERP/6666	EPA 7471	MERC/6640
60129815006	BHSM06/SS 0-0.5	EPA 7471	MERP/6666	EPA 7471	MERC/6640
60129815007	BHSM07/SS 0-0.5	EPA 7471	MERP/6666	EPA 7471	MERC/6640
60129815008	BHSM08/SS 0-0.5	EPA 7471	MERP/6666	EPA 7471	MERC/6640
60129815009	BHSM08A/SS 0-0.5	EPA 7471	MERP/6666	EPA 7471	MERC/6640
60129815010	BHSM09/SS 0-0.5	EPA 7471	MERP/6666	EPA 7471	MERC/6640
60129815011	BHSM10/SS 0-0.5	EPA 7471	MERP/6666	EPA 7471	MERC/6640
60129815012	BHSM11/SS 0-0.5	EPA 7471	MERP/6666	EPA 7471	MERC/6640
60129815013	BHSM12/SS 0-0.5	EPA 7471	MERP/6666	EPA 7471	MERC/6640
60129815014	BHSM13/SS 0-0.5	EPA 7471	MERP/6666	EPA 7471	MERC/6640
60129815001	BHSM01/SS 0-0.5	ASTM D2974	PMST/7789		
60129815002	BHSM02/SS 0-0.5	ASTM D2974	PMST/7789		
60129815003	BHSM03/SS 0-0.5	ASTM D2974	PMST/7789		
60129815004	BHSM04/SS 0-0.5	ASTM D2974	PMST/7789		
60129815005	BHSM05/SS 0-0.5	ASTM D2974	PMST/7789		
60129815006	BHSM06/SS 0-0.5	ASTM D2974	PMST/7789		
60129815007	BHSM07/SS 0-0.5	ASTM D2974	PMST/7789		
60129815008	BHSM08/SS 0-0.5	ASTM D2974	PMST/7789		
60129815009	BHSM08A/SS 0-0.5	ASTM D2974	PMST/7789		
60129815010	BHSM09/SS 0-0.5	ASTM D2974	PMST/7789		
60129815011	BHSM10/SS 0-0.5	ASTM D2974	PMST/7789		
60129815012	BHSM11/SS 0-0.5	ASTM D2974	PMST/7789		
60129815013	BHSM12/SS 0-0.5	ASTM D2974	PMST/7789		
60129815014	BHSM13/SS 0-0.5	ASTM D2974	PMST/7789		

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CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Page: 1 of 2

1545158

Section A Required Client Information:

Company: Burns & McDonnell
Address: 9400 Ward Parkway
Kansas City, Mo 64114
Email To: sshelton@burnsmcd.com
Phone: 816-333-9400 Fax:
Requested Due Date/TAT: Std

Section B Required Project Information:

Report To: Sharon Shelton
Copy To: Daniel Earhart
Purchase Order No.:
Project Name: Boiler House
Project Number: 68635

Section C Invoice Information:

Attention: Sharon Shelton
Company Name: Burns & McDonnell
Address:
Pace Quote Reference:
Pace Project Manager:
Pace Profile #:

REGULATORY AGENCY

☐ NPDES ☐ GROUND WATER ☐ DRINKING WATER
☐ UST ☐ RCRA ☐ OTHER

Site Location STATE: MO

Section D Required Client Information

SAMPLE ID (A-Z, 0-9 / ,-) Sample IDs MUST BE UNIQUE

Matrix Codes MATRIX / CODE

Drinking Water	DW
Water	WT
Waste Water	WW
Product	P
Soil/Solid	SL
Oil	OL
Wipe	WP
Air	AR
Tissue	TS
Other	OT

Requested Analysis Filtered (Y/N)

ITEM #	SAMPLE ID	MATRIX CODE (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED COMPOSITE START DATE	COMPOSITE START TIME	COMPOSITE END/GRAB DATE	COMPOSITE END/GRAB TIME	SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Analysis Test Y/N	Analysis Test: 7471 Mercury	Residual Chlorine (Y/N)	Pace Project No./ Lab I.D.
1	BHSM 01 /SS 0-0.5	SL	G			9-26-12	955		1	X								X	X		001
2	BHSM 02 /SS 0-0.5	SL	G			9-26-12	1010		1	X								X	X		002
3	BHSM 03 /SS 0-0.5	SL	G			9-26-12	1015		1	X								X	X		003
4	BHSM 04 /SS 0-0.5	SL	G			9-26-12	1020		1	X								X	X		004
5	BHSM 05 /SS 0-0.5	SL	G			9-26-12	1025		1	X								X	X		005
6	BHSM 06 /SS 0-0.5	SL	G			9-26-12	1035		1	X								X	X		006
7	BHSM 07 /SS 0-0.5	SL	G			9-26-12	1040		1	X								X	X		007
8	BHSM 08 /SS 0-0.5	SL	G			9-26-12	1045		1	X								X	X		008
9	BHSM 08A /SS 0-0.5	SL	G			9-26-12	—		1	X								X	X		018
10	BHSM 09 /SS 0-0.5	SL	G			9-26-12	1050		1	X								X	X		009
11	BHSM 10 /SS 0-0.5	SL	G			9-26-12	1055		1	X								X	X		011
12	BHSM 10 MS/MSD /SS 0-0.5	SL	G			9-26-12	1055		1	X								X	X		MS/MSD

1 UNPRES
2 UNPRES

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME
Retain samples following analysis, and contact Sharon Shelton prior to disposal,		9-26-12	1350		9/26/12	1356

SAMPLE CONDITIONS

Temp in °C	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)
4.0	Y	N	Y

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER: Daniel Earhart Brian Hoye
SIGNATURE of SAMPLER:
DATE Signed (MM/DD/YY): 9-26-12

ORIGINAL

*Important Note: By signing this form you are accepting Pace's NET 30 day payment terms and agreeing to late charges of 1.5% per month for any invoices not paid within 30 days.

F-ALL-Q-020rev.07, 15-May-2007

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CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

1545159

Section A Required Client Information:

Company:	Burns & McDonnell
Address:	9400 Ward Parkway
	Kansas City, MO 64114
Email To:	sshelton@burnsmcd.com
Phone:	816-333-9400
Fax:	
Requested Due Date/TAT:	STD

Section B Required Project Information:

Report To:	Sharon Shelton
Copy To:	Daniel Earhart
Purchase Order No.:	
Project Name:	Bala House
Project Number:	62635

Section C Invoice Information:

Attention:	Sharon Shelton
Company Name:	Burns & McDonnell
Address:	
Pace Quote Reference:	
Pace Project Manager:	
Pace Profile #:	

REGULATORY AGENCY

☐ NPDES ☐ GROUND WATER ☐ DRINKING WATER
☐ UST ☐ RCRA ☐ OTHER

Site Location STATE: MO

Section D Required Client Information

Matrix Codes MATRIX / CODE

Drinking Water	DW
Water	WT
Waste Water	WW
Product	P
Soil/Solid	SL
Oil	OL
Wipe	WP
Air	AR
Tissue	TS
Other	OT

SAMPLE ID
(A-Z, 0-9 / ,-)
Sample IDs MUST BE UNIQUE

Requested Analysis Filtered (Y/N)

ITEM #	SAMPLE ID	MATRIX CODE (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED: COMPOSITE START DATE	COMPOSITE START TIME	COMPOSITE END/GRAB DATE	COMPOSITE END/GRAB TIME	SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Analysis Test Y/N: Mercury 7471	Residual Chlorine (Y/N)	Pace Project No./ Lab I.D.
1	BHSM 11 /SS0-0.5	SL	G			9-26-12	1100		1	X								X		60129815 012
2	BHSM 12 /SS 0-0.5	SL	G			9-26-12	1105		1	X								X		013
3	BHSM 13 /SS 0-0.5	SL	G			9-26-12	1110		1	X								X		014
4																				
5																				
6																				
7																				
8																				
9																				
10																				
11																				
12																				

1 WGFU

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS			
Retain Samples following analysis and contact Sharon Shelton prior to disposal.		9-26-12	1350		9/26/12	1355	4-8	Y	N	Y

ORIGINAL

SAMPLER NAME AND SIGNATURE

PRINT Name of SAMPLER: Daniel Earhart Brian Hoye

SIGNATURE of SAMPLER: DATE Signed (MM/DD/YY): 9-26-12

Temp in °C	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)

*Important Note: By signing this form you are accepting Pace's NET 30 day payment terms and agreeing to late charges of 1.5% per month for any invoices not paid within 30 days.

Sample Condition Upon Receipt

Pace Analytical® www.pacelabs.com

Client Name: Burns & McDonnell Project # 60129815

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☑ Client ☐ Commercial ☐ Pace ☐ Other ____

Tracking #: ____ Pace Shipping Label Used? ☐ Yes ☑ No

Custody Seal on Cooler/Box Present: ☐ Yes ☑ No Seals intact: ☐ Yes ☑ No

Optional
Proj. Due Date: 10/8
Proj. Name: Boiler House

Packing Material: ☐ Bubble Wrap ☑ Bubble Bags ☐ Foam ☐ None ☐ Other ____

Thermometer Used: T-191 / T-194 Type of Ice: Wet Blue None ☐ Samples on ice, cooling process has begun

Cooler Temperature: 4-8

Temperature should be above freezing to 6°C

Date and Initials of person examining contents: 9/26 LS

		Comments:
Chain of Custody present:	☑Yes ☐No ☐N/A	1.
Chain of Custody filled out:	☑Yes ☐No ☐N/A	2.
Chain of Custody relinquished:	☑Yes ☐No ☐N/A	3.
Sampler name & signature on COC:	☑Yes ☐No ☐N/A	4.
Samples arrived within holding time:	☑Yes ☐No ☐N/A	5.
Short Hold Time analyses (<72hr):	☐Yes ☑No ☐N/A	6.
Rush Turn Around Time requested:	☐Yes ☑No ☐N/A	7.
Sufficient volume:	☑Yes ☐No ☐N/A	8.
Correct containers used:	☑Yes ☐No ☐N/A	9.
-Pace containers used:	☑Yes ☐No ☐N/A	
Containers intact:	☑Yes ☐No ☐N/A	10.
Unpreserved 5035A soils frozen w/in 48hrs?	☐Yes ☐No ☑N/A	11.
Filtered volume received for dissolved tests	☐Yes ☐No ☑N/A	12.
Sample labels match COC:	☑Yes ☐No ☐N/A	13.
-Includes date/time/ID/analyses Matrix: SL		
All containers needing preservation have been checked.	☐Yes ☐No ☑N/A	14.
All containers needing preservation are found to be in compliance with EPA recommendation.	☐Yes ☐No ☑N/A	
Exceptions: VOA, coliform, TOC, O&G, WI-DRO (water), Phenolics	☐Yes ☑No	Initial when completed ____ Lot # of added preservative ____
Trip Blank present:	☐Yes ☐No ☑N/A	15.
Pace Trip Blank lot # (if purchased): ____		
Headspace in VOA vials (>6mm):	☐Yes ☐No ☑N/A	16.
Project sampled in USDA Regulated Area:	☐Yes ☐No ☑N/A	17. List State: ____

Client Notification/ Resolution: Copy COC to Client? Y / N Field Data Required? Y / N

Person Contacted: ____ Date/Time: ____

Comments/ Resolution: ____

Project Manager Review: AKR for AKR Date: 9/28/12

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of hold, incorrect preservative, out of temp, incorrect containers)

APPNEDIX F
Mercury Speciation Analysis

BROOKS RAND LABS
MEANINGFUL METALS DATA

Summary and Comparison of Brooks Rand Labs SOP #BR-0013 (5-Step Selective Sequential Extraction Procedure for Mercury Speciation in Solids) and EPA Method 3200

BR-0013 ("The 5-Step Procedure") Summary

SOP #BR-0013 is a performance-based procedure followed at Brooks Rand Labs to determine the mercury levels in sediments by using a five-step selective sequential extraction procedure.

Fraction	Description	Extractant	typical compounds
F1	water soluble	DI water	$HgCl_2$ $HgSO_4$
F2	weak acid soluble/"stomach acid"	pH 2 HCl/HOAc	HgO
F3	organo complexed	1M KOH	Hg-humics Hg_2Cl_2 CH_3Hg
F4	strong complexed	12M HNO_3	mineral lattice Hg_2Cl_2 Hg^0
F5	mineral bound/cinnabar	aqua regia	HgS m-HgS HgSe HgAu

The ability to generate data for the total level of metals in sediments is well known and can be achieved by a variety of different technologies. While this can be useful in a broad sense it is often more important to gain an understanding of either the individual species present or the mobility of the material present in the matrix. This SOP deals with the use of a five-step extraction procedure to determine the mercury level in sediments.

Mercury is extracted from a accurately weighed sediment sample into five different solutions, these can be broadly linked to types of mercury compounds. The extractants used are: deionized water, a synthetic "stomach acid", 1 M potassium hydroxide solution, 12 M nitric acid, and aqua regia. After extraction, samples are analyzed by EPA Method 1631 (CVAFS).

Mercury species in each fraction has been demonstrated to selectively extract the following mercury compounds and/or species.

BROOKS RAND LABS

MEANINGFUL METALS DATA

EPA Method 3200 Summary

1.0 SCOPE AND APPLICATION

1.1 This method contains a sequential extraction and separation procedure that may be used in conjunction with a determinative method to differentiate mercury species that are present in soils and sediments. This method provides information on both total mercury and various mercury species.

1.2 The speciation of a metal, in this case mercury, involves determining the actual form of the molecules or ions that are present in the sample. When combined with an appropriate determinative method, this procedure is designed to provide varying degrees of mercury species information. All metal speciation methods are operationally defined by the level of post-extraction processing and the chosen method of analysis. Examples of the operationally-defined mercury fractions and individual species that may be determined using this procedure are presented in the table below.

The environmental mobility and toxicity of mercury in a soil profile depend on its speciation. Alkyl mercury species such as methylmercury are at least an order of magnitude more mobile than inorganic mercury species, and thus are more toxic and more readily bioaccumulated. Soluble inorganic mercury species such as mercury chloride are more easily transported by natural process than the other inorganic mercury species and serve as the substrate for mercury methylation process (Ref. 1). These extractable organomercury species and extractable inorganic species contribute the major portion of mercury potential toxicity in the soils. The mercury species that fall into the "semi-mobile" category such as elemental mercury are less toxic than extractable mercury species. The "non-mobile" mercury species such as mercury sulfide are chemically stable in the soil for geologic time periods and thus are least toxic.

1.3 Quantification of mercury in the different fractions may be performed using any suitable technique with appropriate precision and accuracy, for example Method 7473, 1631, or Methods 7470 and 7471. Other analytical techniques, such as gas chromatography-mass spectrometry (GC-MS), ion chromatography or high performance liquid chromatography (HPLC) with either GC-MS or inductively coupled plasma-mass spectrometry (ICP-MS) detection (Method 6020), or other hyphenated and/or mass spectrometric techniques, may be employed if performance appropriate for the intended application can be demonstrated. This method may also be applicable to other matrices, such as industrial and municipal waste materials, but its performance on such matrices has not yet been evaluated. Method 6800 (Elemental and Speciated Isotope Dilution Mass Spectrometry) (Ref. 2) may also be applicable as a diagnostic and validation tool for quantification of selectively extracted mercury species, especially when species transformations occur in the sample preparation or analysis procedures.

Operationally-Defined Mercury Fractions		Individual Mercury Species	CAS No.[a]
Total Mercury			
Extractable Mercury	Extractable Organic Mercury	CH_3HgCl	115-09-3
		CH_3CH_2HgCl	107-27-7
	Extractable Inorganic Mercury	$HgCl_2$	7487-94-7
		$Hg(OH)_2$	[b]—
		$Hg(NO_3)_2$	10045-94-0
		$HgSO_4$	13766-44-4
		HgO	21908-53-2
		Hg^{2+} complexes[c]	—
Non-extractable Mercury	Semi-mobile Mercury	Hg^0	7439-97-6
		Hg^0-M[d]	—
		Hg^{2+} complexes[c]	—
		Hg_2Cl_2 (minor)	10112-91-1
	Non-mobile Mercury	Hg_2Cl_2 (major)	10112-91-1
		HgS	1344-48-5
		HgSe	20601-83-6

[a]Chemical Abstract Service Registry Number
[b]Not registered by the Chemical Abstract Service
[c]Certain inorganic mercury complexes may be present in both the organic and inorganic extractable fractions
[d]This represents a mercury-metal amalgam

BROOKS RAND LABS

MEANINGFUL METALS DATA

Comparison

The 5-step procedure is usually viewed as being more obviously applicable to environmental samples. The extractants used include regular water and a weak acid, which can be more applicable to bioavailability work. Method 3200 requires sample sonication, heating of the extracts, and the use of more complex reagents.

Method 3200 method detection limits (MDLs) are all equal or lower than the MDLs achieved with the 5-step procedure. Method 3200 may be more selective for organic Hg compounds in the Extractable Organic Mercury phase than the F3 extract of the 5-step procedure. The Extractable Inorganic Mercury phase of Method 3200 combines the first two steps of the 5-step procedure.

Brooks Rand Labs prices for these two tests are equivalent if all extractions/steps are requested.

3958 6th Ave NW • Seattle, WA 98107 • T: 206-632-6206 • F: 206-632-6017 • www.brooksrand.com • brl@brooksrand.com

SOP #BR-0013

Five-Step Selective Sequential Extraction Procedure (SEP) to Quantify Mercury Fractions in Sediments, Soils and Mine Tailings

Brooks Rand Labs

Revision 002
Written 04/12/04
Revised 1/19/09

Reviewed _____
_____
_____
_____

VP of Analytical Services	2/17/09 Date
QA Manager	2/17/09 Date
Scientist (if applicable)	2-17-09 Date

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Five-Step Selective Sequential Extraction Procedure (SEP) to Quantify Mercury Fractions in Sediments, Soils and Mine Tailings

1.0 SCOPE AND APPLICATION

SOP-0013 is a performance-based procedure followed at Brooks Rand LLC to determine the mercury levels in sediments by using a five-step selective sequential extraction procedure.

2.0 SUMMARY OF METHOD

2.1 The ability to generate data for the total level of metals in sediments is well known and can be achieved by a variety of different technologies. While this can be useful in a broad sense it is often more important to gain an understanding of either the individual species present or the mobility of the material present in the matrix. This SOP will deal with the use of a five-step extraction procedure to determine the mercury level in sediments.

2.2 Mercury is extracted from a accurately weighed sediment sample into five different solutions, these can be broadly linked to types of mercury compounds. The extractants used are: deionized water, a synthetic "stomach acid", 1M potassium hydroxide solution, 12M nitric acid and aqua regia. After extraction, samples are analyzed by EPA Method 1631 (Brooks Rand SOP #BR-0006).

2.3 Mercury species in each fraction has been demonstrated (see references) to selectively extract the following mercury compounds and/or species.

Fraction	Description	Extractant	typical compounds
F1	water soluble	DI water	$HgCl_2$ $HgSO_4$
F2	weak acid soluble/"stomach acid"	pH 2 HCl/HOAc	HgO
F3	organo complexed	1M KOH	Hg-humics Hg_2Cl_2 CH_3Hg
F4	strong complexed	12M HNO_3	mineral lattice Hg_2Cl_2 Hg^0
F5	mineral bound/cinnabar	aqua regia	HgS m-HgS HgSe HgAu

3.0 INTERFERENCES

All Brooks Rand Labs (BRL) SOPs are Proprietary Information and protected by WA state law. Proprietary Information shall be kept in the strictest confidence & shall not be used or appropriated to benefit any party without prior written consent from BRL.

9.0 QUALITY CONTROL

Table 1. Method Quality Assurance Criteria and Frequency.

QC Sample	Measure	Minimum Frequency	Criteria	Corrective Action
Bubbler Blank	Contamination from bubblers	1 per bubbler used prior to analysis	each ≤ 40 pg avg ≤ 20 pg std ≤7.5 pg	Clean and test bubblers until criteria met prior to any analysis
Calibration Standards	Acceptability of the Calibration Curve	Whenever > 48 hours since last batch analyzed using the calib. or OPR/QCS fail	RSD of response factors ≤ 15%; Recovery of Low Standard = 80 – 120%	Reanalyze suspect calibration standard. If criteria still not met, then remake standards and recalibrate the instrument
Continuing Calibration Verification (CCV)	Accuracy	2 per batch (one at the beginning and one at the end of each batch)	Recovery = 77 – 123%	Correct problem and reanalyze CCV. If criteria met, reanalyze samples backwards until 2 consecutive results w/RPD ≤ 20%
Independent Calibration Verification (ICV)	Accuracy	1 per batch immediately following the calibration	Recovery = 85 – 115%	Correct problem and reanalyze ICV. Recalibrate instrument if necessary.
Carryover Check Bubbler Blank	Contamination due to carryover in the bubbler/trap	On same bubbler/trap following any result exceeding ½ the carryover threshold of 100,000 pg	≤ 50 pg and within ± 20 pg of avg bubbler blank	Clean and continue to test bubbler/trap combo until criteria met prior to further use. Samples analyzed following a result ≥ ½ the carryover threshold must be reanalyzed
Method Blank	Contamination from reagents, lab ware, etc.	3 per batch	F1 - MB < 0.2 F2 - MB < 0.2 F3 - MB < 2 F4 - MB < 2 F5 - MB < 2 or High MB < 1/10th of associated samples	Correct problem until criteria met. All samples associated with a contaminated method blank must be reanalyzed.
Quality Control Sample (QCS) equiv. to CRM/SRM	Accuracy	1 per batch	Recovery = 75 – 125% from sum of results of 5 Hg fractions	Correct problem prior to continuing analysis
Method Duplicate	Precision within a given matrix	1 per 10 client samples	RPD ≤ 35%	If **RPD** criteria not met, then the system is not in control. Correct problem and reanalyze all associated samples.

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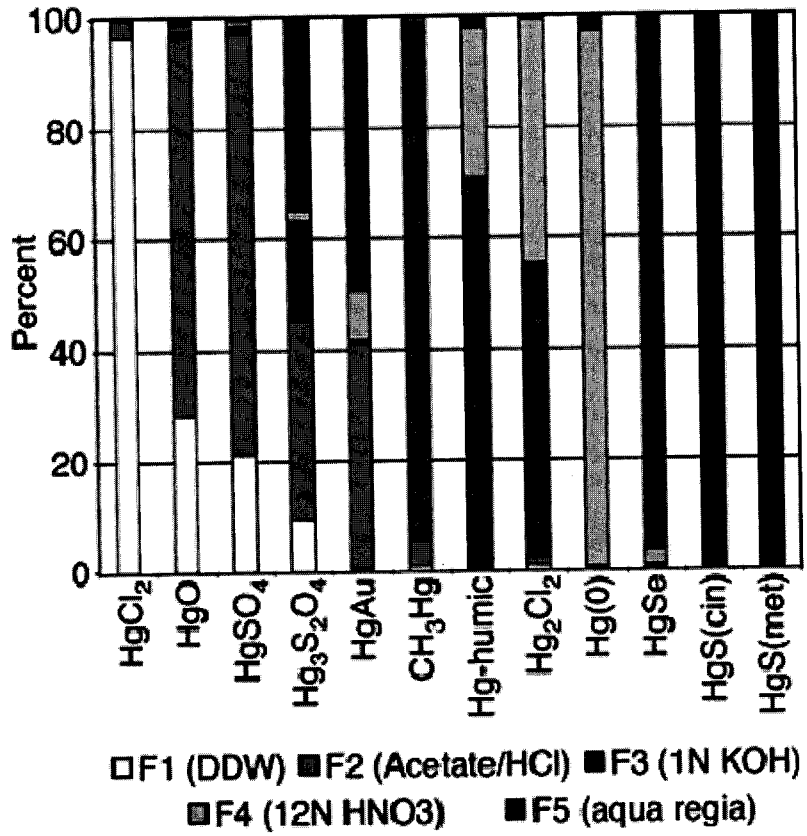

Figure 1. Extraction fingerprint showing the percent of total mercury extracted in each fraction for 10 mercury compounds suspended in kaolin. These compounds are typically used as reference materials during this analysis. The concentration of mercury (ug/g) in each compound is as follows: HgSe, 1.02; HgAu, 0.1; Hg2Cl2, 6.7; HgSO4, 4.70; HgS, 4.3; *m*-HgS, 9.6; Hg0, 44.2; HgO, 3.6; HgCl2, 2.6; Hg–humic, 0.05; CH3Hg, 0.02 ng/g. Derived from Bloom, N.S. et al (2003).

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10.0 REFERENCES

Bloom, N.S., Preus, E., Katon, J., and Hiltner, M. (2003). "Selective extractions to assess the biogeochemically relevant fractionation of inorganic mercury in sediments and soils", *Analytica Chimica Acta* 479: 233-248.

Brooks Rand SOP #BR-0006. "BRL procedure for EPA Method 1631: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry."

Brooks Rand SOP #BR-0012. "Determination of "acid-labile mercury and mercury sulfide in solids by Cold Vapor Atomic Fluorescence Spectrometry (CVAFS)."

EPA Method 1631 Revision E. (2002). "Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry."

EPA Method 3200 Draft 2 (2003). "Mercury species by selective solvent extraction and acid digestion"

Liang, L. and Bloom, N.S. (1992). "Determination of Total Hg by Single-Stage Gold Amalgamation with Cold Vapor Atomic Spectrometric Detection." *JAAS.* 8:001

Revis, N.W. et al. (1989). "Quantitative Method for Determining the Concentration of Mercury(II) Sulfide in Soils and Sediments." *Analyst* 114: 823-825.

Sahuquillo, A., Rauret, G., Bianchi, M., Rehnert, A., and Muntau, H. (2003). "Mercury determination in solid phases from application of the modified BCR-sequential extraction procedure: a valuable tool for assessing its mobility in sediments", *Anal Bioanal Chem* 375: 578-583.

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An Overview of Techniques for Mercury Speciation of Contaminated Sediments

Annie Carter and Michelle L. Briscoe
Brooks Rand Labs, Seattle WA USA

Introduction

Mercury is often considered an environmental pollutant of interest because of its potential toxic effects on humans and the environment(1). Mercury contamination can occur through a variety of natural and anthropogenic processes. The fate and transport of mercury in the environment (Figure 1) can vary greatly depending on the environmental substrates to which it is complexed or its chemical speciation(2–6). Though total mercury analysis is now commonplace, it is often useful to determine the dominant form of mercury in order to successfully remediate or better characterize the environmental hazards of a specific site(7). In soils and sediments, often only a very small portion of the total mercury is bioavailable(2,3). Though there is no universally-accepted method for measuring mercury lability in the environment, a variety of methods exist in the literature to determine operationally-defined mercury fractions in a sample(1–4,6,8,9). This analysis is particularly useful for contaminated mine wastes that may contain high levels of mercury, but have low bioavailability because the mercury is primarily in a stable form such as mercury sulfide(1–4,6,10). A fraction refers to a group of compounds that have similar characteristics, such as bioavailability or environmental mobility, but cannot be distinguished from one another

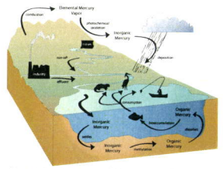

Figure 1: Fate & Transport of Mercury in the Environment

Thermal Volatilization

Thermal volatilization, or thermodesorption, coupled to an atomic absorption detector (TDAAS) is a technique that has been employed for the determination of mercury species. TDAAS works by heating a sample at a specific rate and temperature. As the sample is heated, the different mercury compounds are released at different temperature ranges(6,12). As the mercury is released, it is swept into an atomic absorption spectrophotometer and the absorbance is recorded on a chromatogram. The species of mercury is determined by comparing the thermodesorption profile to that of a known, pure source(6,12). This technique may produce peaks that do not fit well with any of the known models due to the complex interactions and oxidation states of mercury compounds(6). Though it is a good method for determining elemental mercury and gaining information about mercury oxidation states, thermal volatilization does not provide much information about mercury mobility and bioavailability(12).

X-Ray Absorption Fine Structure Spectroscopy (XAFS)

X-Ray Absorption Fine Structure Spectroscopy (XAFS) uses high energy X-rays from a synchrotron source to identify species based on their scattering pattern(5,6). While this technique can give detailed information about oxidation states and compound structures, it is limited in usefulness as the total mercury concentration in a sample must be greater than 100 µg/g(4,5). Additionally, the mercury model compound database does not include all of the possible species of mercury, potentially resulting in the detection of unknown species(4,5). In an independent experiment, Bloom and Christopher S. Kim compared results of the SSE procedure (see next section) to XAFS for the reference materials provided by SGC (see *Reference Materials* section). The HgS and HgSe species identified by XAFS agreed well with the fraction recovered in Fraction 5 of the SSE procedure(4). However, the more soluble mercury species such as HgO, as identified by XAFS, were recovered partially or fully in Fraction 4 of the SSE, rather than in Fraction 2, as the SSE model projected(4). This could be due to the mercury-containing particles being encapsulated within larger particles or could be spectral misidentification from XAFS(4).

References

1. Horvat, M. *et al.* Remediation of Mercury Polluted Sites Due to Mining Activities. *Critical Reviews in Analytical Chemistry* **33**, 291–296 (2003).
2. Bloom, N. S., Preus, E., Katon, J. & Hiltner, M. Selective extractions to assess the biogeochemically relevant fractionation of inorganic mercury in sediments and soils. *Analytica Chimica Acta* **479**, 233–248 (2003).
3. Shi, J., Liang, L., Jiang, G. & Jin, X. The speciation and bioavailability of mercury in sediments of Haihe River, China. *Environment international* **31**, 357–365 (2005).
4. Kim, C. S., Bloom, N. S., Rytuba, J. J. & Brown Jr, G. E. Mercury speciation by X-ray absorption fine structure spectroscopy and sequential chemical extractions: a comparison of speciation methods. *Environmental science & technology* **37**, 5102–5108 (2003).
5. Kim, C. S., Rytuba, J. J. & others Characterization and speciation of mercury-bearing mine wastes using X-ray absorption spectroscopy. *The Science of the total environment* **261**, 157–168 (2000).
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5-Step Selective Sequential Extraction (SSE) Procedure

The SSE procedure was developed specifically for mercury and its known unique physical and chemical properties(2). It separates total mercury into five different fractions based on behavioral classes(2). The sum of fractions 1-3 is considered to be total bioavailable mercury(2,3). Fractions 4 and 5 represent the semi-mobile and non-mobile fractions respectively. The SSE sample preparation procedure consists of leaching a pre-weighed aliquot of sediment with a series of continuously stronger reagents (Figure 2). The supernatants from each leaching step are analyzed for total mercury concentration by stannous chloride reduction, nitrogen purging onto a gold sand trap, and thermal desorption into a cold vapor atomic fluorescence detector following the protocol described in EPA Method 1631 Appendix(7).

Some of the advantages of the SSE are the low detection limits, which allow potentially contaminated sites to be compared to background values. Additionally, SSE provides the ability to determine with some confidence the potential contribution from elemental mercury(2). The SSE method was developed by a renowned mercury researcher and verified by a number of labs with a history of mercury analysis but is not an accepted EPA method.

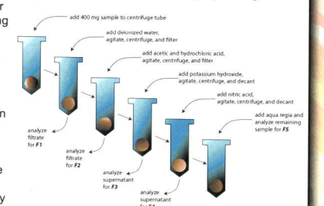

Figure 2: Overview of 5-Step SSE Procedure

EPA Method 3200

Method 3200 is an accepted EPA method for the determination of mercury fractions. As with the SSE, it is an operationally-defined procedure and does not provide specific mercury species information but rather provides informational values for a group of species. For Method 3200, there is an initial preparation procedure that yields a supernatant and a pellet (Figure 3). The supernatant then is further separated into extractable organic and extractable inorganic fractions. The pellet is further separated into semi-mobile and non-mobile fractions(11).

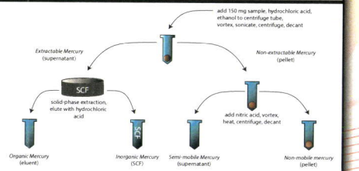

Figure 3: Overview of EPA Method 3200

Comparison of 5-Step SSE and EPA Method 3200

Table 1 compares some of the advantages and disadvantages of the SSE procedure and Method 3200. They are both operationally defined procedures that provide information on the distribution of mercury into different fractions that can help determine what remediation methods may be the most useful. Depending on what the requirements are for a certain project, one method may be more applicable than the other.

5 Step SSE	EPA Method 3200
sequential sample preparation	semi-sequential sample preparation
straightforward reagents that make sense biogeochemically	multiple reagents and steps that are pH dependent
does not require specialized equipment	requires *Sulphydryl Cotton Fiber*, solid phase extraction system, and sonication heating source or laboratory microwave
can provide information on the presence of elemental mercury	does not distinguish elemental mercury from other species
published in peer-reviewed journals and validated by labs in a round robin study	EPA method

Table 1

EPA 3200 Fractions		*mercury species*	SSE Fractions
Extractable	Inorganic	$HgCl_2$	F1 – water soluble
		$HgSO_4$	
		HgO	F2 – weak acid soluble
		$Hg(OH)_2$	
		$Hg(NO_3)_2$	
	Organic	Hg-humics	F3 – organo complexed
		CH_3HgCl	
		CH_3CH_2HgCl	
Non-extractable	Semi-mobile	Hg_2Cl_2	F4 – strong complexed
		Hg^0	
		Hg^{++} complexes	
	Non-mobile	HgS	F5 – mineral bound
		HgSe	
		HgAu	

Table 2

Table 2 shows the relative similarities in fractionation between Method 3200 and the SSE procedure. Fractions 1 and 2 of the SSE procedure are expected to be roughly equivalent to the extractable inorganic mercury portion of Method 3200. Fraction 3 of the SSE is equivalent to the extractable organic portion of Method 3200 except Hg_2Cl_2 is recovered in Fraction 3 for the SSE compared to being recovered in the semi-mobile and non-mobile fractions for 3200. Fraction 4 of the SSE is expected to be equivalent to the semi-mobile fraction. Fraction 5 of the SSE is expected to be equivalent to non-mobile for Method 3200.

Reference Materials

No certified reference materials are commercially available for the mercury fractionation methods. However, SSE reference materials were developed by Nicolas S. Bloom, formerly of *Studio Geochimica* (SGC), for the purpose of method validation. These reference materials (for Hg^0, HgS, and $HgCl_2$) were made by dispersing pure Hg compounds in kaolin clay(2). These reference materials, along with some real world samples prepared by SGC, were distributed to eight labs who participated in a round robin study in 2005 to validate different analytical methods for the speciation of mercury in solid samples. The results were presented by Bloom at the *International Conference on Mercury as a Global Pollutant* in Madison, Wisconsin, in 2006. Though these reference materials are not officially certified for any analyte, SGC provided expected concentrations for each fraction of the SSE procedure, as well as for total mercury. National Institute of Standards and Technology (NIST) Standard Reference Material 2710 is *Montana Soil* that has a total mercury concentration of 32.6 mg/kg.

NIST 2710 and the SGC reference materials (Hg^0, HgS, and $HgCl_2$) were prepared by EPA Method 3200 and by the 5-Step SSE.

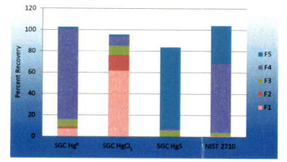

Figure 4: SSE Recoveries of Reference Materials

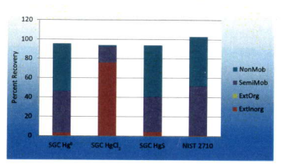

Figure 5: Method 3200 Recoveries of Reference Materials

Figure 4 shows the result of each fraction of the SSE procedure as a percentage of the sum of the mercury fractions. The sum of all fractions then shows the recovery of the reference material as a percentage of the certified or reference value.

Figure 5 shows the result of each fraction of the Method 3200 procedure as a percentage of the sum of the mercury fractions. The sum of all fractions then shows the recovery of the reference material as a percentage of the certified or reference value.

Figure 6 shows the percent of each reference material that would be considered bioavailable by each method. For the SSE, fractions 1-3 were summed. For Method 3200, the Extractable Inorganic and Extractable Organic fractions were summed (there was no contribution from the extractable organic mercury fraction in this case).

Figure 7 compares the Semi-mobile fraction from Method 3200 to Fraction 4 of the SSE. Figure 8 compares the Non-mobile fraction from Method 3200 to Fraction 5 of the SSE.

The semi-mobile and non-mobile fractions generally do not compare well with fractions 4 and 5 from the SSE. According to the method, elemental mercury should be extracted in the semi-mobile fraction, but when the SGC reference material for Hg^0, a material made by dispersing elemental mercury in kaolin clay, was analyzed, a significant portion (49%) was extracted in the non-mobile fraction. For the SSE procedure, 85% of mercury in the SGC Hg^0 reference material was extracted in Fraction 4, as would be expected(2,11). Similarly, for the SGC HgS reference material, only 53% was recovered in the non-mobile fraction for Method 3200, while 76% was recovered in Fraction 4 for SSE. HgS is primarily expected to be recovered in the non-mobile fraction, though there may be some more mobile forms(11).

For both the SSE procedure and Method 3200 analyses, analyzing total mercury in the sample provides an important quality assurance check. This allows for a comparison of the sum of the fractions to the total mercury concentration. In this way, sample loss, contamination, or other errors can be assessed if the sum of fractions is significantly different from the total mercury concentrations(2). It also allows for a more appropriate interpretation of the data: because the fractions are operationally-defined, having a known concentration of a certain fraction is not very meaningful by itself, rather it needs to be taken in the context of the sample as a whole(2,11).

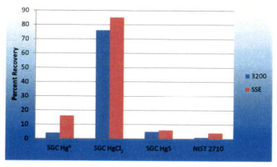

Figure 6: Bioavailable Mercury in Reference Materials

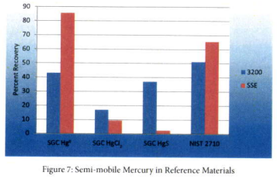

Figure 7: Semi-mobile Mercury in Reference Materials

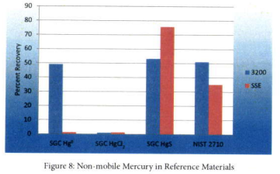

Figure 8: Non-mobile Mercury in Reference Materials